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*Indian Standard*

METHODS OF SAMPLING AND TEST FOR  
RESINS FOR PAINTS

**PART 1 GENERAL TEST METHODS**

*( Second Revision )*

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**BUREAU OF INDIAN STANDARDS**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

# *Indian Standard*

## METHODS OF SAMPLING AND TEST FOR RESINS FOR PAINTS

### PART 1 GENERAL TEST METHODS

### *(Second Revision)*

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# *Indian Standard*

## METHODS OF SAMPLING AND TEST FOR RESINS FOR PAINTS

### PART 1 GENERAL TEST METHODS

### *(Second Revision)*

## 0. FOREWORD

**0.1** This Indian Standard ( Part 1 ) ( Second Revision ) was adopted by the Indian Standards Institution on 1 January 1987, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

**0.2** This standard was originally published in 1952 to cover methods of sampling and general test methods mainly for natural resins. Subsequently, an Indian Standard for methods of sampling and test for natural and synthetic resins was published as Part 2 of the standard in 1971. These two parts were amalgamated on revision in 1976. The present revision has been necessitated as more and more newer synthetic resins like polyamides, polyvinyls, chlorinated rubber and emulsion polymers are being manufactured and used in the country. While revising the standard, the Committee responsible for the preparation of this standard felt it appropriate to publish this standard in various parts, as indicated below:

Part 1 General test methods

Part 2 Special test methods for alkyd resins

Part 3 Special test methods for phenolic resins

Part 4 Special test methods for epoxy resins

Part 5 Special test methods for polyamide resins

Part 6 Special test methods for amino resins

Part 7 Special test for determination of monomer content in acrylic or vinylacetate containing polymers and emulsions

Part 8 Special test methods for chlorinated rubber



**0.3** In this standard, test methods covered in 2 to 16 and 17.8 of IS : 354-1976\* have been included. In addition to above, methods of test for colour by Gardner colour scale, viscosity by Brookfield viscometer and Stormer viscometer, softening point by capillary tube, non-volatile matter and flash point have been added.

**0.4** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

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## 1. SCOPE

**1.1** This standard ( Part 1 ) prescribes the methods of sampling and general test for resins ( natural and synthetic ) used in paints, enamels and varnishes.

## 2. TERMINOLOGY

**2.1** For the purpose of this standard, the definitions given in IS : 1303-1963‡ and IS : 6667-1972§ shall apply.

## 3. SAMPLING

**3.1 General Requirements** — In drawing and storing samples, the following precautions and directions shall be observed.

**3.1.1** Samples shall not be taken in an exposed place.

**3.1.2** Sampling instruments shall be clean and dry at the commencement of sampling.

**3.1.3** The sampled material shall be placed in clean, dry and air-tight containers on which the material has no action.

**3.1.4** Each sample container shall be sealed after filling and marked with full details of sampling.

### 3.2 Scale of Sampling

**3.2.1 Lot** — All the containers in a consignment of resins of the same type and same batch of manufacture shall be grouped to constitute a lot.

---

\*Methods of sampling and test for resins for paints ( *first revision* ).

†Rules for rounding off numerical values ( *revised* ).

‡Glossary of terms relating to paints ( *revised* ).

§Glossary of terms used in synthetic resin industry.

3.2.2 The number of containers to be selected for sampling shall be as given in Table 1.

TABLE 1 SCALE OF SAMPLING

NO. OF CONTAINERS IN THE LOT	NO. OF CONTAINERS TO BE SELECTED FOR SAMPLING
Up to 50	3
51 to 100	4
101 to 200	5
201 to 300	6
301 and above	7

3.2.2.1 These containers shall be selected at random from the lot, preferably with the help of random number tables ( see IS : 4905-1968\* ).

### 3.3 Procedure for Taking Samples from Containers

3.3.1 *Solid Resins* — If the material is supplied in the form of large lumps packed in containers, a suitable sampling implement is required to withdraw samples from the selected containers. It is recommended that a portable steel spike, with a wooden handle, as shown in Fig. 1, be used. The total mass of the spike shall be 2 kg and the mass of the steel shank shall be 1.5 kg.

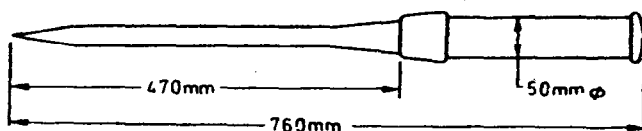


FIG. 1 STEEL SPIKE

3.3.1.1 From each of the selected containers, remove the top surface to a depth of 10 cm with the help of steel spike. From the exposed surface draw a sample of 500 g of resin at different depths using the steel spike. The collected material shall be broken to small lumps and kept in separate sample containers. These individual samples representing each of the selected containers in the lot shall be examined for colour and visible impurities.

\*Methods for random sampling.

**3.3.1.2** From each of the individual samples, approximately equal quantities of resin shall be taken and mixed together to give a composite sample weighing 1 kg. This composite sample shall be used for conducting various tests mentioned in material specifications.

**3.3.2 Resin Solution** — If the resin is supplied in the form of solution, each of the containers selected from the lot shall be opened and individually examined for clarity, sedimentation/settling and layer separation. After these visual tests are conducted, approximately equal quantities of resin solution shall be taken from the selected containers at different depths for checking solids and viscosity in order to ascertain the homogeneity of solution and these portions shall be mixed together to give a composite sample of 1 litre.

### 3.4 Number of Tests and Criteria for Conformity

**3.4.1** Tests for colour, visible impurities and, if required by the purchaser, test for relative density shall be conducted on each of the individual samples. Tests for all other characteristics shall be conducted on the composite sample.

**3.4.2** The lot shall be deemed to conform to the specification if all the test results ( *see* 3.4.1 ) satisfy the corresponding requirements when tested as prescribed in this standard.

## 4. QUALITY OF REAGENTS

**4.1** Unless specified otherwise, pure chemicals and distilled water ( *see* IS : 1070-1977\* ) shall be used.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

## 5. PRELIMINARY EXAMINATION

**5.1** The original sealed sample container shall be opened and condition of the contents noted, paying particular attention to the presence of foreign and visible impurities.

## 6. COLOUR

**6.0 Outline of the Method** — The colour of the resins or resin solutions shall be determined by (a) Lovibond tintometer; or (b) Gardner colour scale.

---

\*Specification for water for general laboratory use ( *second revision* ).

**6.0.1** In Lovibond tintometer, the colour of resins is determined by comparison with Lovibond glasses of known colour characteristics. The colour is expressed as the sum total of the yellow and red slides used to match the colour of the resin in a cell of the specified size in the Lovibond tintometer. In Gardner colour scales, the comparison of the colour of a sample, in a glass tube of standard diameter, with the colours of arbitrarily numbered colour standards is carried out for identification of the standard that most closely matches the colour of the sample and result is expressed in terms of a Gardner colour number.

## **6.1 Lovibond Tintometer Method**

### **6.1.1 Apparatus**

#### **6.1.1.1 Lovibond tintometer**

#### **6.1.1.2 Glass cells — 1, 2, 5 and 10 cm.**

**6.1.2 Procedure** — Dissolve a known mass of the solid resin in an equal mass of a suitable colourless solvent; in case the resin is in solution form, fill the same in a clean dry glass cell of desired size as such or diluted to fifty percent, if required. Place the cell in position in the tintometer and compare the colour of the solution or resin with the combination of glass slides through an eye piece.

**6.1.3 Report** — Report the colour of the resin or resin solution in terms of Lovibond units and also state dilution percentage, if applicable, as follows:

Colour reading in cell ( size and designation of the glass cell used, to be given ) = (  $a Y + b R$  )

where

$a$  = the sum total of the various yellow ( Y ) slides used, and

$b$  = the sum total of the various red ( R ) slides used.

## **6.2 Gardner Colour Scale Method**

### **6.2.1 Apparatus**

#### **6.2.1.1 Gardner colour standards**

- a) *Reference standards* — Reference standards are glass colour standards having chromaticity coordinates and luminous transmittances as specified in Table 2. Eighteen glass colour standards are required. The glass colour standards shall be checked by the method given below:

Select a dual beam spectrophotometer with a sufficiently small light beam at the sample position so that all rays will pass through the standard to be calibrated. Alternatively equip the spectrophotometer with a condensing lens to achieve this. Place the standards in turn in the sample position of the spectrophotometer. If the comparator is provided with a separate green filter in front of the light source, place this filter in the reference beam of the dual beam spectrophotometer during calibration of each standard. Obtain spectral transmittance data for each glass reference standard. From the spectral transmittance data for each reference standard, calculate the *CIE* tristimulus values,  $X$ ,  $Y$ ,  $Z$ , and the chromaticity coordinates  $x$  and  $y$  for *CIE* illuminant  $C$ .

- b) *Working standards* — Working standards are 18 glass or liquid colour standards having chromaticity coordinates that differ from those of the reference standards by not more than one-third of the difference in  $x$  or  $y$  ( see Table 2 ) between adjacent reference standards. In any one set, no two standards shall be closer together than two-thirds of the difference in  $x$  or  $y$  between corresponding reference standards. The luminous transmittances shall be as specified in Table 2. The liquid working standards are freshly prepared coloured solutions contained in glass test tubes. Potassium chloroplatinate solutions are used for the lighter standards ( 1 to 8 ), and solutions of ferric chloride and cobalt chloride in hydrochloric acid are used for the darker standards ( 9 to 18 ).
- c) The compositions of the liquid colour standards prepared are as follows:
  - i) *Gardner colour standards 1 to 8* — Into each of a series of one-mark volumetric flasks of the capacities indicated in Table 3, transfer from a burette the corresponding volumes of potassium hexachloroplatinate solution ( see Note 4 ) as shown in Table 3, dilute each to the mark with the hydrochloric acid solution ( see Note 3 ) and mix well.
  - ii) *Gardner colour standards 9 to 18* — Into a series of 100-ml one-mark volumetric flasks, transfer from burettes the volumes of the ferric chloride solution ( see Note 5 ) and cobalt chloride solution ( see Note 6 ) as shown in Table 4. Dilute each to the mark with the hydrochloric acid solution ( see Note 3 ) and mix well.

NOTE 1 — In case of dispute, only glass reference standards shall be used.

NOTE 2 — The chromaticity coordinates of the glass standards and those of the liquid standards differ slightly. However, the difference does not affect the accuracy of the estimation.

NOTE 3 — Hydrochloric acid ( 1 : 17 ) solution. Mix one volume of concentrated hydrochloric acid ( specific gravity approximately 1.19 g/ml ) with 17 volumes of water.

NOTE 4 — Potassium hexachloroplatinate solutions. Dissolve 790 mg of potassium hexachloroplatinate (  $K_2PtCl_6$  ) in the hydrochloric acid solution ( *see* Note 3 ) in a 100-ml one-mark volumetric flask. Warm the solution until all the potassium hexachloroplatinate is dissolved. Cool to 20°C room temperature, dilute to the mark with the same hydrochloric acid solution and mix well.

NOTE 5 — Cobalt chloride solution. Dissolve 40 g of cobalt chloride hexahydrate (  $CoCl_2 \cdot 6H_2O$  ) in 120 g of the hydrochloric acid solution ( *see* Note 3 ).

NOTE 6 — Ferric chloride solution. Dissolve 1 000 g of ferric chloride hexahydrate (  $FeCl_3 \cdot 6H_2O$  ) in 240 g of hydrochloric acid solution ( *see* Note 1 ). Heat gently if necessary. Adjust the concentration so that the solution has exactly the same colour as a freshly prepared 30 g/l solution of potassium dichromate in concentrated sulphuric acid ( specific gravity approximately 1.84 g/ml ).

**6.2.1.2 Glass test tubes** — clear, colourless, round, of inside diameter  $10.65 \pm 0.025$  mm, outside diameter about 12.5 mm and outside length about 114 mm.

NOTE — Test tubes that approximate to the stated diameter and that are, preferably, not less than 10 mm or not more than 11 mm inside diameter may be used. In such cases, the results should be multiplied by a correction factor equal to  $10.65/d$ , where  $d$  is the inside diameter of the test tube.

**6.2.1.3 Colour comparator** — constructed to illuminate uniformly, and to permit simultaneously visual comparison of light transmitted through two colour standards and through a test tube in a transverse direction.

The apparatus may be of any design but should have the following characteristics:

- a) *Illumination* — CIE Illuminant C.
- b) *Surrounding field* — The field should not differ significantly in brightness from the samples and standards and shall be essentially achromatic.
- c) *Field of view* — Two standards and a sample shall always be in the field of view.
- d) *Arrangement of standard and sample* — There shall be a perceptible gap between sample and standard, but this shall be as small as possible.

**6.2.2 Procedure** — Fill a clean test tube ( 6.2.1.2 ) to a height of at least 70 mm with the sample, passing it through a sintered glass filter if there is any visual turbidity. Place the sample tube in the sample compartment of the comparator ( 6.2.1.3 ). Switch on the light source and

compare simultaneously the colour of the sample with the colours of two adjacent standards, at a viewing distance between 30 and 50 cm. Determine which standard most closely matches the sample in brightness and saturation, ignoring any differences of hue.

### 6.2.3 Expression of Results

**6.2.3.1 Reporting** — Report the number of the standard most closely matching the colour of the sample. If more precise measurements are needed, report as either lighter than, matching or darker than the standard and expressed as a Gardner Colour number and also state the type of standard used (that is), glass colour standard or liquid colour standard.

TABLE 2 COLOUR SPECIFICATIONS OF REFERENCE STANDARDS

[ Clause 6.2.1.1 (a) and (b) ]

SL No.	GARDNER COLOUR STANDARD NUMBER	CHROMATICITY COORDINATES		LUMINOUS TRANSMI- TTANCE Y, PERCENT	TRANSMIT- TANCE TOLERANCE ( $\pm$ )
		X	Y		
(1)	(2)	(3)	(4)	(5)	(6)
1	1	0.317 7	0.330 3	80	7
2	2	0.323 3	0.335 2	79	7
3	3	0.332 9	0.345 2	76	6
4	4	0.343 7	0.364 4	75	5
5	5	0.355 8	0.384 0	74	4
6	6	0.376 7	0.406 1	71	4
7	7	0.404 4	0.435 2	67	4
8	8	0.420 7	0.449 8	64	4
9	9	0.434 3	0.464 0	61	4
10	10	0.450 3	0.476 0	57	4
11	11	0.484 2	0.481 8	45	4
12	12	0.507 7	0.463 8	36	5
13	13	0.539 2	0.445 8	30	6
14	14	0.564 6	0.427 0	22	6
15	15	0.585 7	0.408 9	16	2
16	16	0.604 7	0.392 1	11	1
17	17	0.629 0	0.370 1	6	1
18	18	0.647 7	0.352 1	4	1

**TABLE 3 COMPOSITION OF POTASSIUM HEXACHLOROPLATINATE SOLUTIONS****( Gardner Colour Standards 1 to 8 )***[ Clause 6.2.1.1 ( c ) ( i ) ]*

GARDNER COLOUR STANDARD NUMBER	POTASSIUM HEXACHLOROPLATINATE SOLUTION ml	NOMINAL CAPACITY OF THE ONE-MARK VOLUMETRIC FLASK ml
(1)	(2)	(3)
1	3.48	50
2	5.47	50
3	8.42	50
4	6.58	25
5	9.60	25
6	5.35	10
7	8.10	10
8	10.00	10

**TABLE 4 COMPOSITION OF IRON AND COBALT SOLUTIONS****( Gardner Colour Standards 9 to 18 )***[ Clause 6.2.1.1 ( c ) ( ii ) ]*

GARDNER COLOUR STANDARD NUMBER	FERRIC CHLORIDE SOLUTION ml	COBALT ( II ) CHLORIDE SOLUTION ml	HYDROCHLORIC ACID SOLU- TION ml
(1)	(2)	(3)	(4)
9	3.8	3.0	93.2
10	5.1	3.6	91.3
11	7.5	5.3	87.2
12	10.8	7.6	81.6
13	16.6	10.0	73.4
14	22.2	13.3	64.5
15	29.4	17.6	53.0
16	37.8	22.8	39.4
17	51.3	25.6	23.1
18	100.0	0.0	0.0



## 7. RELATIVE DENSITY

**7.0 Outline of the Method** — This method prescribes the determination of relative density of materials which are highly viscous, solids or semi-solids and free-flowing liquids at 27°C, by pyknometers.

### 7.1 Apparatus

**7.1.1 Pyknometer** — conforming to Type 4 ( for free-flowing liquids ) and Type 6 ( for viscous, semi-solid or solid materials ) of IS : 5717-1970\* as shown in Fig. 2.

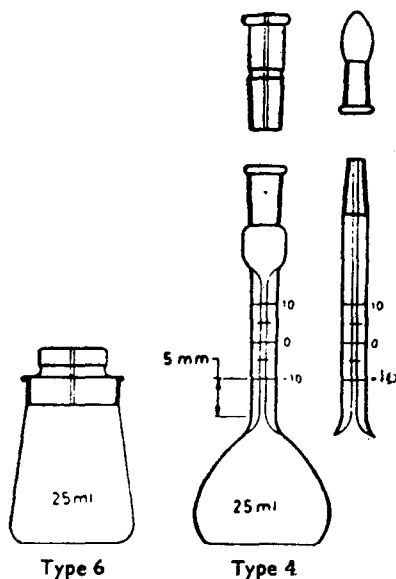


FIG. 2 PYKNOMETERS

### 7.2 Procedure

**7.2.1 Calibration** — Clean the pyknometer first by soaking in potassium chromate solution and then washing thoroughly with water, alcohol and ether. Dry the pyknometer thoroughly and weigh accurately. Fill the pyknometer with freshly boiled water, taking care that no air-bubbles

\*Specification for pyknometers.

are entrapped. Keep the pyknometer in a water-bath maintained at  $27 \pm 2^\circ\text{C}$  till constant temperature is attained ( approximately 30 minutes ). Remove the pyknometer from bath, wipe dry with a clean cloth and weigh immediately.

**7.2.2 For Liquids that Flow Readily** — Fill a clean, dry pyknometer with the material to be tested, proceed as prescribed in 7.2.1 and record the mass. Calculate the relative density of the material as the ratio of the mass of the material and equal volume of water.

NOTE — If air-bubbles are entrapped, centrifuge the material either before or after filling the pyknometer.

**7.2.3 For Viscous, Semi-Solid or Solid Materials** — Fill a clean, dry pyknometer one-half full with the material to be tested. Precautions shall be taken to prevent the material from touching the sides of the pyknometer above the final level and to prevent inclusion of air-bubbles. It is advisable to warm the bottle before filling. Cool the bottle with its contents to  $27 \pm 2^\circ\text{C}$  in a water-bath. Remove the pyknometer, wipe dry and weigh immediately. Fill the pyknometer with water and quickly push under water contained in a beaker at  $20^\circ\text{C}$ . Insert the stopper while the pyknometer is under water and transfer the pyknometer to a water-bath maintained at  $27 \pm 2^\circ\text{C}$ . Allow the pyknometer to attain the temperature of bath ( approximately 30 minutes ). At the end of this period, carefully blot dry the top of the stopper, being careful not to draw water from stopper opening. Remove the pyknometer from bath, wipe dry and weigh.

**7.2.3.1 Calculation** — Calculate relative density as follows:

$$\text{Relative density} = \frac{M_3 - M_1}{(M_2 - M_1) - (M_4 - M_3)}$$

where

$M_3$  = mass in g of the pyknometer plus material,

$M_1$  = mass in g of empty pyknometer,

$M_2$  = mass in g of the pyknometer plus water, and

$M_4$  = mass in g of the pyknometer plus material plus water.

## 8. VISCOSITY

**8.0 Outline of the Method** — Viscosity of resin solutions may be determined by : (a) bubble tube method, or (b) flow cup method, or (c) Ostwald U-tube method, or (d) Brookfield viscometer, or (e) Stormer viscometer.

**8.0.1** In bubble tube method, the resin is dissolved in solvent and the bubble seconds are determined. In the flow cup method, the relative consistency of the sample is measured by a simple control test under specified conditions and is expressed in terms of flow seconds. Ostwald U-tube method measures viscosity in terms of flow seconds. Brookfield method measures viscosity in terms of Poises. In Stormer method the consistency of the resins is measured by means of the load required to produce a rotational frequency of 200 rpm for an offset paddle rotor immersed in a viscous medium.

## **8.1 Bubble Tube Method**

### **8.1.1 Apparatus**

**8.1.1.1 Bath** — a constant temperature bath.

**8.1.1.2 Holder for viscosity tubes** — Preferably a mechanical holder capable of holding the tube in exact vertical position after being placed in constant temperature bath.

**8.1.1.3 Mechanical shaker**

**8.1.1.4 Timing device** — suitable timing device like stop-watch capable of reading to a precision of 0.1 second.

**8.1.1.5 Viscosity tubes** — of clean glass and flat bottom, having  $10.650 \pm 0.025$  mm inside diameter and  $114 \pm 1$  mm length and having circular markings at  $27.0 \pm 0.5$ ,  $100.0 \pm 0.5$  and  $108.0 \pm 0.5$  mm from the bottom outside the tube.

**8.1.1.6** When kinematic viscosity is to be determined directly, viscosity tubes of the following dimensions and details shall be used:

Flat bottom, open mouth glass tube having inside diameter of  $11.50 \pm 0.25$  mm and length  $101.50 \pm 1.25$  mm. It is fitted with a cork so that the distance from the bottom of the cork to the bottom of the tube is  $90.0 \pm 1.0$  mm. The tube is to be filled with the material under test up to a height so that the air space left will give a bubble of  $15.0 \pm 1.0$  mm length when it will ascend through the material.

### **8.1.2 Reagent**

**8.1.2.1 Solvent** — as agreed to between the purchaser and the supplier,

### **8.1.3 Procedure**

**8.1.3.1 Preparation of resin solutions** — The procedure employed for dissolving solid resins will depend upon the chemical nature of resin. The solution may be obtained either by cold cut or hot cut method as agreed

to between the purchaser and the supplier. The two procedures should not be interchanged:

- a) *Cold-cut method* — Weigh a 250-ml screw cap bottle with a cellulose film (100 × 100 mm) to nearest 0.05 g. Weigh into the bottle the required amount of solvent (40 g for a 60 percent solution). Weigh in a beaker the required amount of resin (60 g for 60 percent solution). Transfer the resin slowly into the bottle containing solvent, swirl gently to wet. Place the cellulose film over the mouth of the bottle and screw cap tightly. Shake the flask in the shaker overnight. Check the mass of the bottle and solution at the completion of shaking. If the loss is appreciable, discard solution and prepare fresh one.
- b) *Hot-cut method* — Weigh accurately a long necked 250-ml flask with ground stopper. Weigh into the flask the required amount of solvent and resin. Connect the flask to a suitable air or water condenser and gently warm over a suitable oil or liquid paraffin bath with occasional swirling to prevent any charring. Usually the solution will be complete in about 15 minutes. When solution is complete, cool the flask, disconnect and weigh. If the loss of solvent is over 0.1 g, add the necessary amount of solvent to compensate loss, swirl and mix.

NOTE — If any air bubbles are present, allow the solution to stand until they disappear.

8.1.3.2 The resins supplied as solutions may be employed as such for determination of viscosity. However, if dilution is necessary, such as reduction to a specified solids content, use a suitable solvent as agreed to between the purchaser and the supplier.

8.1.3.3 Place the flask containing the resin solution in the bath at  $27 \pm 2^\circ\text{C}$ . Fill a viscosity tube with the solution exactly to 100-mm mark, taking care to avoid any of the sample on the tube wall above 108-mm mark. Insert a new cork stopper so that the bottom of the cork is level with the 108-mm mark. This will give a bubble of uniform size and reproducible pressure. Insert the tube in the holder with stoppered-end down, and place the assembly in the bath at  $27 \pm 2^\circ\text{C}$  for about 10 to 15 minutes to attain temperature equilibrium. With the assembly still in the bath, push the holder to return the tube to upright position and start the time device when the top of the bubble becomes tangent with the 100 mm line. Record the time when the bubble again becomes tangent to 27-mm mark to nearest 0.1 second. Record at least three determinations which agree within 1.0 second. Report the average of these values as result.

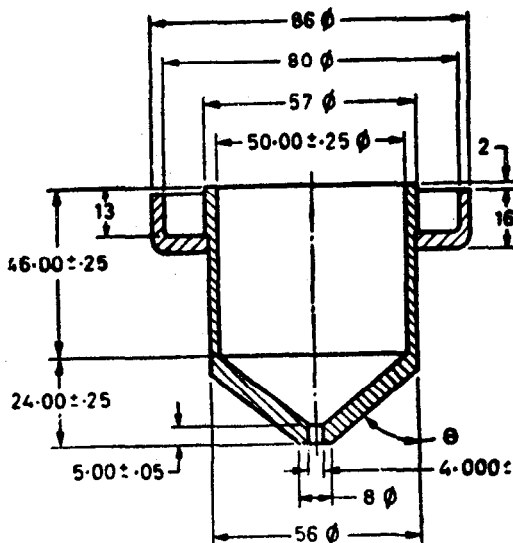
**8.1.3.4 Report** — Report the 73-mm bubble travel time in seconds as viscosity at  $27 \pm 2^\circ\text{C}$ , and report solution concentration, solvent used and method of making the solution

**NOTE** — When kinematic viscosity is required, it shall be obtained by multiplying time obtained in seconds using a tube described in 8.1.1.6 by a factor 1.2.

## 8.2 Flow Cup Method

**8.2.1 Flow Cup** — The necessity in industry for measuring the relative consistency by a simple control test during manufacture and use of paints, varnishes, lacquers and other viscous products has led to the use of flow cups. The cup ( Generally known as Ford Cup Viscometer No. 4. ) described in this standard gives only an approximate measure of consistency as distinct from a measure of viscosity, the cup flow timing being influenced by the amount of mechanical disturbance of the sample before and during the test and is not accurate for very viscous materials.

**8.2.2 Form and Dimensions** — The flow cup shall be essentially of the form and dimensions shown in Fig. 3.



All dimensions in millimetres.

FIG. 3 FLOW CUP

**8.2.3 Material of Construction** — A cup made of any non-ferrous material is suitable. This may be plated. The finish shall be smooth.

**8.2.3.1** The jet may be either bored directly or constructed separately of stainless steel and force-fitted. Care is essential in order to avoid damage to the lower apex of the cup. A protective skirt, which does not interfere with flow, may be provided.

**8.2.4** The following apparatus shall be used in carrying out a test:

- a) A thermometer accurate to within  $0.5^{\circ}\text{C}$ ;
- b) A stop-watch or stop-clock;
- c) A suitable stand, provided with levelling screws;
- d) A spirit level; and
- e) A straight-edged scraper for the top of the cup.

**8.2.5 Procedure** — Place the flow cup on the stand in a place free from draught, preferably with the air temperature within the range  $30 \pm 5^{\circ}\text{C}$ . Level by the use of a spirit level placed on the rim.

**8.2.5.1** Strain the sample into a clean container and adjust the temperature to meet the requirements specified in 8.2.5. A 150-micron IS Sieve [ see IS : 460 ( Part 2 )-1985\* ] or finer is suitable. This and the following operations shall be carried out with minimum delay to avoid loss of solvent:

- a) With the orifice closed by the finger, fill the cup with the bubble-free sample until it just begins to overflow into the gallery, pouring slowly to minimize the formation of air bubbles. If bubbles are present, allow them to rise and then remove them from the surface.
- b) Check that the temperature of the material in the cup is within  $0.5^{\circ}\text{C}$  of the test temperature. The cup may be at a temperature different from that of the sample and it is recommended that a minute or so be allowed to elapse before checking the temperature.
- c) Place the scraper on the rim of the cup and draw it firmly across until the excess of the sample has flowed into the gallery. Place the receiver under the cup. Remove the finger and simultaneously start the stop-watch. Watch the stream of liquid flowing from the orifice. At the first evidence of a break of the stream into droplets, stop the stop-watch. The time taken is recorded in seconds as time of flow in flow cup.

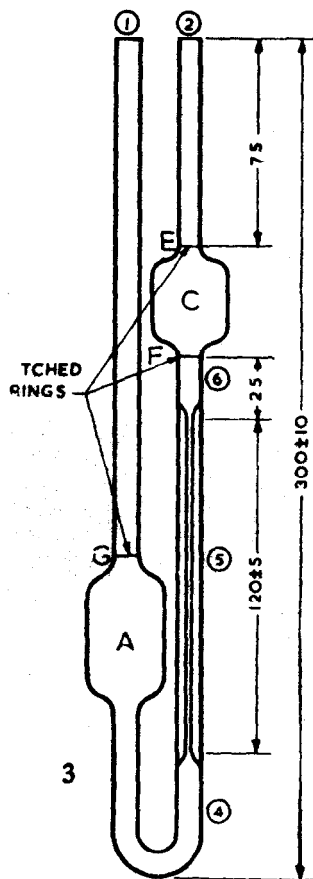
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\*Specification for test sieves : Part 2 Perforated plate test sieves ( *third revision* ).

### 8.3 Ostwald U-Tube Viscometer Method

#### 8.3.1 Apparatus

**8.3.1.1 U-tube viscometer** — The U-tube viscometer shall be of homogeneous transparent glass and free from mechanical imperfections. All glass tubing employed in the construction of the viscometer shall be of the same composition and the finished instrument shall be thoroughly annealed. The design, size and the dimensions of the viscometer to be used for various viscosity ranges are shown in Fig. 4 and Table 5.



All dimensions in millimetres.

FIG. 4 OSTWALD U-TUBE VISCOMETER

**8.3.1.2 Thermometer** — Any convenient thermometer of suitable range with 0.1 to 0.2°C subdivisions.

**8.3.1.3 Bath** — A water-bath suitable for immersion of viscometer within 5 cm from the top with provision for visibility of the instrument and the thermometer shall be provided. The viscometer may be fixed as an integral part of the bath. The thermometer shall be mounted with the bulb near the centre of the viscometer. The bath shall be regulated so that the variation in the thermometer reading does not exceed 0.2°C. Use of thermo-regulator is recommended.

**8.3.1.4 Time recording device** — A suitable stop-watch.

**8.3.1.5 Frame** — Suitable frame shall be provided so that the viscometer may be suspended securely in the bath in a vertical position.

### 8.3.2 Procedure

**8.3.2.1** Select an appropriate U-tube viscometer and clean it by rinsing with suitable solvents, such as benzene ( *see* IS 534-1974\* ), followed by ethyl ether ( *see* IS : 336-1973† ) or petroleum hydrocarbon solvents ( *see* IS : 1745-1978‡ ). Remove each solvent by passing a current of dry air through it and take care that no moisture remains inside the instrument. Periodically or whenever necessary, clean the instrument by use of chromic acid cleaning solution, allowing it to stand in the instrument for several hours. After cleaning, rinse the viscometer with water, clean and dry with the solvents as given above.

**8.3.2.2** Suspend the cleaned viscometer in the bath maintained at  $27 \pm 0.2^\circ\text{C}$ , taking care to ensure that the capillary arm is vertical. Fill the viscometer through tube 1 with the material, using a long pipette to minimize any wetting of the tube above the filling mark *G*. Allow at least 20 minutes for the viscometer to reach the test temperature and adjust the volume to bring the liquid within 0.2 mm of the filling mark. After the sample has reached the test temperature, apply suction or pressure to bring the liquid level up to a point one centimetre above the timing mark *E*. Release the suction or pressure and measure the time required for the bottom of the meniscus to pass from the top edge of mark *E* to the top edge of mark *F*.

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\*Specification for benzene ( *second revision* ).

†Specification for ether ( *second revision* ).

‡Specification for petroleum hydrocarbon solvents ( *second revision* ).



TABLE 5 DIMENSIONS OF U-TUBE VISCOMETER

( Clause 8.3.1.1, and Fig. 4 )

i) *Size	B	C	D
ii) Viscosity range, cS	4 to 10	7.5 to 35	20 to 120
iii) Tubes 1, 3 and 4, mm	8 to 9 OD and 1 to 1.25 wall		9 to 10 OD and 1 to 1.25 wall
iv) Bulb A:			
a) Made from tubing, mm	21 to 23 OD and 1 to 1.25 wall		25 to 27 OD and 1 to 1.25 wall
b) Length of parallel sides, Min, mm	20	20	28
v) Bore of capillary 5, $\pm 0.01$ mm	0.71	0.88	1.4
vi) Tubes 2 and 6, mm	6 to 7 OD and 1 to 1.25 wall		7 to 8 OD and 1 to 1.25 wall
vii) Bulb C:			
a) Made from tubing, mm	21 to 23 OD and 1 to 1.5 wall		25 to 27 OD and 1 to 1.5 wall
b) Capacity, ml	5.0 $\pm$ 0.25	5.0 $\pm$ 0.25	10.0 $\pm$ 0.5
viii) Vertical distance FG, mm	87 $\pm$ 4	83 $\pm$ 4	78 $\pm$ 4
ix) Distance between centres of tubes 1 and 2, mm	20	20	20

\*See IS : 1448 [ P : 25 ] - 1976 Methods of test for petroleum and its products: [ P : 25 ] Determination of kinetic and dynamic viscosity ( *first revision* ).

### 8.3.3 Calculation

$$V = C_t - \frac{B}{t}$$

where

$V$  = kinematic viscosity in centistokes;

$C_t$  = constant for the instrument ( *see 8.3.4* );

$B$  = viscometer coefficient, the kinetic energy correction; and  
 $t$  = time in seconds.

**8.3.3.1** The coefficient kinetic energy for the test viscometer shall be calculated as follows:

$$B = \frac{t_1 \times t_2}{t_2^2 - t_1^2} (V_2 t_1 - V_1 t_2)$$

where

$t_1$  = efflux time ( 200 seconds, *Min* ) for an oil having a kinematic viscosity of  $V_1$ ; and

$t_2$  = efflux time for an oil having a kinematic viscosity of  $V_2$  ( about five times greater than  $V_1$  ).

**NOTE** — With viscometer of constant  $C$  greater than 0.05 and with a minimum efflux time of 200 seconds,  $B/t$  is not greater than 0.1 percent of the total, and may be neglected; then the equation in 8.3.3 becomes  $V = C/t$ .

### 8.3.4 Determination of Viscometer Constant $C$

**8.3.4.1** Unless the constant is already known, **calibrate the viscometer** using primary standard, namely, oil samples the viscosities of which have been determined by the National Physical Laboratory, New Delhi, or any other institution recognized by the Government of India.

**8.3.4.2** Determine, in the viscometer being calibrated, the efflux time of the appropriate standard which shall not be less than 200 seconds by the method as prescribed in 8.3.2. Then calculate the constant  $C$  as follows by substituting the efflux time  $t$  and the kinematic viscosity  $V$  of the oil:

$$\text{Viscometer constant, } C = V + \frac{B/t}{t}$$

where

$V$  = kinematic viscosity of the calibrating oil in centistokes,

$B$  = viscometer coefficient ( the kinematic energy correction ),  
and

$t$  = efflux time in seconds.

**NOTE** — With viscometer of constant  $C$  greater than 0.05 and with a minimum efflux time of 200 seconds,  $B/t$  is not greater than 0.1 percent of the total, and may be neglected; then equation in 8.3.4.2 becomes  $C = V/t$ .

## 8.4 Brookfield Viscometer Method

**8.4.0 General** — Resistance offered to the flow of a liquid is quantified into a factor called viscosity. Viscosity of a solution is directly related to molecular weight, percentage solid content in solution, nature of solvent, temperature of measurement, nature of polymer and method of determination. The absolute unit of viscosity is poise. Occasionally, it is also referred in the units of kinematic viscosity, namely, stokes or centistokes.

$$\frac{\text{Viscosity in poise}}{\text{Density}} = \text{Viscosity in stokes}$$

Viscosities are also expressed in units known as Pa.s.

1 Pa.s. = 10 Ps = 1 000 cPs.

**8.4.0.1** Absolute viscosity of liquid materials, solutions both clear and opaque can be measured by this method. Kinematic viscosities obtained by this method are more direct and accurate for Newtonian type of fluids.

### 8.4.1 Apparatus

**8.4.1.1 Brookfield viscometer** — The Brookfield viscometer — Model RVF or suitable other models as agreed to between the purchaser and the supplier. For viscosities between 0 to 2 000 000 cPs as maximum range and 0 to 500 cPs as minimum range. An eight-speed RVF model known as RVT, has maximum and minimum ranges of 0 to 8 000 000 cPs and 0 to 100 cPs.

NOTE — Brookfield viscometer is available from Brookfield Engineering Laboratories Inc., Stoughton, Massachusetts, USA 02072.

**8.4.1.2 Temperature controlled bath** with a variance limit as  $\pm 0.5^{\circ}\text{C}$  using either oil or water.

**8.4.1.3 Thermometer** —  $110^{\circ}\text{C}$  with  $0.5^{\circ}\text{C}$  division.

**8.4.1.4 Glass beaker** — 600 ml of 104 mm diameter.

**8.4.1.5 Glass rod** — useful to stir the material in beaker.

### 8.4.2 Preparing the Sample

**8.4.2.1** Fill the 600 ml beaker to 3/4th its height, with the test sample and keep the same in the constant temperature bath maintained at  $27 \pm 0.2^{\circ}\text{C}$ , so that the test solution layer is below the layer of liquid in the bath. Carefully stir the test material and check its temperature with the thermometer (stirring should not introduce air bubbles in the medium). When the temperature of the test material has reached the desired value, leave it in the bath without stirring for 15 minutes to allow

normalization. Select a suitable spindle for measuring the viscosity, and bring its temperature along with the spindle guard to the measurement temperature. Fix the spindle with the guard to the instrument. Remove the stirring glass rod and thermometer from the material in the beaker.

### **8.4.3 Procedure**

**8.4.3.1** Place the 600 ml beaker with test solution under the viscometer and lower the instrument such that the spindle dips up to the mark given on the spindle for the purpose. Take care to avoid air bubble sticking to the spindle.

**8.4.3.2** Select a proper speed of the motor such that the reading is always in the middle of the dial range. Setting the speed, start the motor and allow the rotation to continue till the needle remains steady on the dial, note the value. After the reading, allow the spindle to rotate for two more cycles and check if the readings obtained correspond with the former. If not, repeat the rotations checking till two successive constant readings are obtained. It is advisable to confirm the readings with two more speeds, one chosen above and another below the one used for the above determination. Note all the three values.

**8.4.3.3** Convert the readings to viscosity values in centipoise ( pascal seconds ) with the aid of conversion table provided with the instrument. The three values obtained as above should not differ at best by more than 1.0 cPs or 1 percent of the full scale.

The value obtained may also be confirmed with other spindles for which speeds are chosen such that the dial readings are in the middle of the scale.

### **8.4.4 Report**

**8.4.4.1** A report on viscosity shall include the following:

- a) Viscosity in centipoise;
- b) Speed of rotation;
- c) Spindle number;
- d) Temperature of the sample to the nearest 0.5°C;
- e) Method of sample preparation;
- f) Model of Brookfield; and
- g) In case of solution, the percentage solid and the solvent.

## 8.5 Stormer Viscometer Method

**8.5.0 General** — This method is useful in measuring consistency of paints and viscosity of resin solutions or liquids and is best suited for measuring the consistency of colloidal dispersion of heterogenous phases. The mass required to produce a rotational frequency of 200 rpm for an offset paddle rotor immersed in a viscous medium is measured by this method. The unit of consistency ( viscosity ) provided by Stormer viscometer is Kreb units ( KU ). This a logarithmic function of load required to produce 200 rpm.

### 8.5.1 Apparatus

**8.5.1.1 Stormer viscometer** — Stormer viscometer with the paddle rotor, made of stainless steel, shall be as shown in Fig. 5 and 6. To obtain 200 rpm rotation, a stroboscopic timer attachment is provided for fast and accurate measurements.

NOTE — Stormer viscometer can be obtained from Gardner Laboratories Inc., 5521 Landy Lane, Bethesda Md 20814.

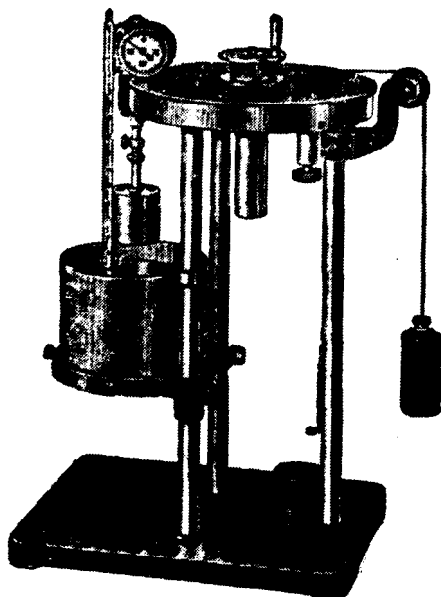
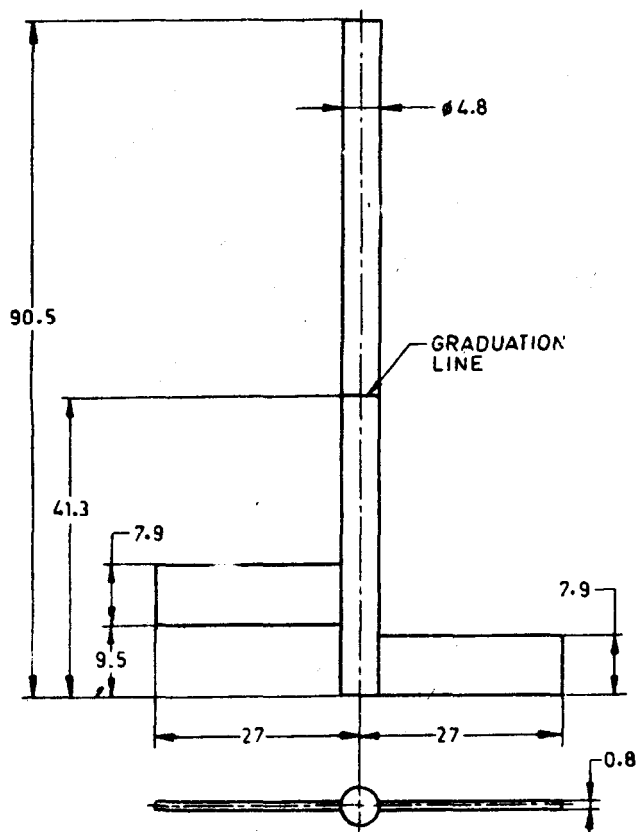


FIG. 5 STORMER VISCOMETER WITH PADDLE-TYPE ROTOR AND STROBOSCOPIC TIMER



All dimensions in millimetres.  
Tolerance on dimensions  $\pm 0.1$  mm.  
Material — Stainless steel.

FIG. 6 PADDLE-TYPE ROTOR FOR USE WITH STORMER VISCOMETER

8.5.1.2 *Beaker* — 500 ml capacity, 85 mm diameter.

8.5.1.3 *Thermometer* —  $110^{\circ}\text{C}$  with  $0.5^{\circ}\text{C}$  divisions.

8.5.1.4 *Weights* — 5-1 000 g.

#### 8.5.2 Calibration of the Instrument

8.5.2.1 Two standard oils, having absolute viscosities (in poise) closer to the material to be measured, are taken and the viscosities of these oils should be at least 5 poise apart.

**8.5.2.2** Assign load values for obtaining 200 rpm to each of the oils by using the equation:

$$L = ( 610\eta + 906 \cdot 6\rho ) / 30$$

where

$\eta$  = viscosity of oil in poise,

$\rho$  = density of the oil, and

$L$  = load.

**8.5.2.3** Make sure the string on the drum is properly wound without overlapping, after removing from the viscometer, the rotor and the weight holder attached to the string.

**8.5.2.4** Place 5 g weight onto the weight holder and release the brake. If the viscometer starts to run from dead start and continues to do so for several windings of string around the drum, then the instrument is quite sensitive. The instrument should be reconditioned, if it does not start.

**8.5.2.5** Dimension of the paddle shall correspond closely to the ones given in Fig. 6 ( tolerance  $\pm 0.1$  mm ).

**8.5.2.6** Standard oil samples, with known viscosities and the load in grams required to produce 200 rpm, are cooled to  $27 \pm 2^\circ\text{C}$ . One of the oils is placed under the instrument with the rotor dipping to the oil up to the mark desired. The exact load required for producing 200 rpm is determined. Similarly, determine the load required for the other oil. If the loads determined are within  $\pm 15$  percent of the calculated loads, the instrument is in satisfactory condition.

### **8.5.3** *Determination of Consistency of Unknown Sample*

#### **8.5.3.1** *Preparation of specimen*

**8.5.3.2** The sample is thoroughly mixed and transferred to a 500 ml beaker to fill it completely leaving 20 mm void space from the top.

**8.5.3.3** It is then placed in a constant temperature water bath to bring the temperature of the sample to  $27 \pm 2^\circ\text{C}$ . Place the sample under the Stormer instrument.

**8.5.3.4** The temperature of the rotor should also be  $27 \pm 0.2^\circ\text{C}$ .

**8.5.3.5** Connect the lamp circuit for the stroboscopic attachment to an electric source.

**8.5.3.6** Connect the weight holder to the string and lower the instrument such that the rotor dips inside the sample to a desired height.

**8.5.3.7** Place weights on the holder and determine the load that will produce 200 rpm pattern on the stroboscopic timer. If the load is more, the lines will look as though they are moving along the rotor direction; reduce the weights in that case. If the lines are moving in the opposite direction then add weights to the holder. The load for 200 rpm will be appearing as stationary lines in the stroboscopic.



FIG. 7 STROBOSCOPIC LINES OPENING WHEN TIMER IS ADJUSTED TO EXACTLY 200 rpm

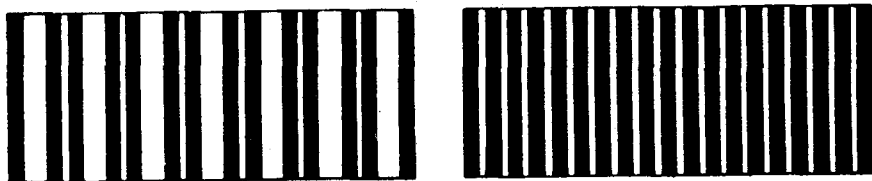


FIG. 8 STROBOSCOPIC LINES APPEARING AS MULTIPLES THAT MAY BE OBSERVED BEFORE 200 rpm IS REACHED

**8.5.3.8** Repeat the test till consistent value of load is obtained ( that is, within 5 g ).

#### **8.5.4 Calculation**

**8.5.4.1** Calculate Kreb units ( KU ) for the load required to produce 200 rpm from the table provided with the instrument.

### **9. REFRACTIVE INDEX**

**9.0 Outline of the Method** — Refractive index is determined with Abbe's refractometer.



## 9.1 Apparatus

**9.1.1 Abbe's Refractometer** — The temperature of the refractometer shall be controlled to within  $\pm 0.1^\circ\text{C}$  and, for this purpose, it shall be provided with a thermostatically controlled water-bath and a suitable device to circulate water through the instrument. The instrument shall be standardized, following the manufacturer's instructions, with a liquid of known purity and refractive index with a glass prism of known refractive index. Distilled water, which has a refractive index of 1.333 0 at  $20.0^\circ\text{C}$  is a satisfactory liquid for standardization.

**9.1.2 Light Source** — If the refractometer is equipped with a compensator, a tungsten lamp or a daylight bulb shall be used. Otherwise, a monochromatic light source, such as an electric sodium vapour lamp shall be used.

**9.2 Procedure** — Filter the material through a filter paper to remove any impurities and traces of moisture. Make sure that the sample is completely dry. Adjust the temperature of the refractometer to  $27.0 \pm 0.1^\circ\text{C}$ . Ensure that the prisms are clean and completely dry, and then place a few drops of the material on the lower prism. Close the prisms, tighten firmly with the screw-head and allow to stand for one or two minutes. Adjust the instrument and light to obtain the most distinct reading possible and determine the refractive index.

## 10. SOFTENING POINT

**10.0 General** — Two methods are prescribed for determination of softening point (a) ring and ball method, and (b) capillary tube method.

### 10.1 Ring and Ball Method

**10.1.0 Outline of the Method** — The sample is enclosed in a horizontal ring and supports a steel ball. The temperature at which the sample sags or flows through a specified distance is the softening temperature.

**10.1.1 Apparatus** — Shall be as follows ( see also Fig. 9 and 10 ).

**10.1.1.1 Ball** — of steel, 9.50 mm in diameter and weighing  $3.50 \pm 0.05$  g.

**10.1.1.2 Ring** — tapered ring as shown in Fig. 10 and of following dimensions:

Depth	$6.35 \pm 0.10$ mm
Inside diameter at bottom	$15.88 \pm 0.10$ mm
Inside diameter at top	$17.46 \pm 0.10$ mm
Outside diameter	$20.60 \pm 0.10$ mm

For convenience of mounting the ring in a support of the type shown in Fig. 9 and 10, the outside diameter of the ring at the bottom may be smaller, but shall not be less than 18.0 mm.

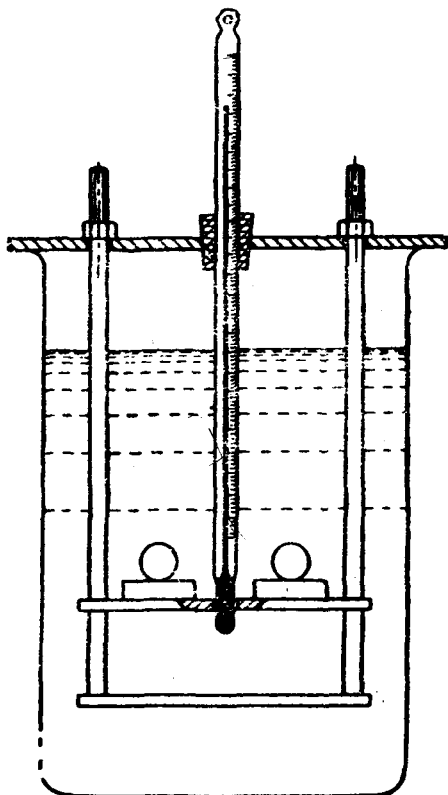


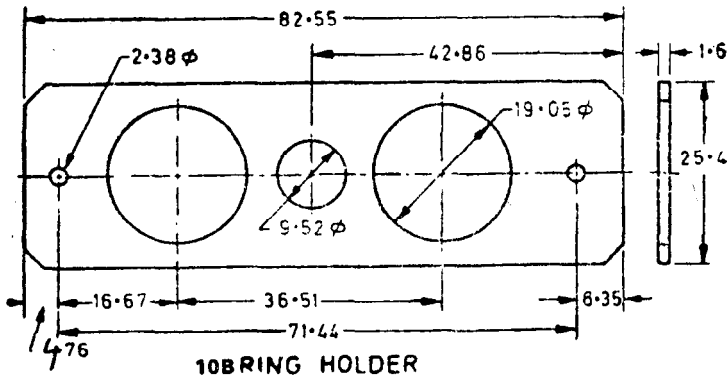
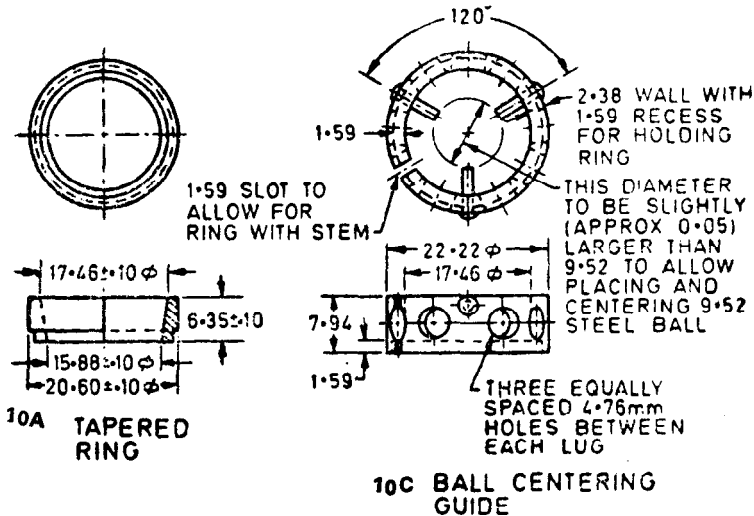
FIG. 9 ASSEMBLY OF APPARATUS FOR DETERMINATION OF SOFTENING POINT ( RING AND BALL — TWO RINGS )

10.1.1.3 *Ball guide* — a convenient form of ball centering guide as shown in Fig. 10.

10.1.1.4 *Support* — a suitable one which fulfils the following conditions:

- a) Rings shall be supported in a horizontal position with the upper surface of the rings 5.0 cm below the surface of the bath liquid;

- b) There shall be a distance of exactly 2.5 cm between the bottom of the rings and the surface of the bottom plate of the support or bottom of the bath; and
- c) The thermometers shall be suspended so that the bottom of the bulb is level with the bottom of the rings, and within 1 cm of the rings, but not touching them.



All dimensions in millimetres.

**FIG. 10 COMPONENTS OF RING AND BALL APPARATUS**

**10.1.1.5 Thermometer** — of range  $-2^{\circ}\text{C}$  to  $+160^{\circ}\text{C}$  with  $0.5^{\circ}\text{C}$  graduations [see IS : 2480 ( Part 1 ) - 1983\* ].

**10.1.1.6 Bath** — made of glass and of suitable size. The bath liquid shall be water for materials having softening point below  $80^{\circ}\text{C}$  and glycerine for materials having softening point above  $80^{\circ}\text{C}$ . But both the liquids shall not be a solvent for the material under test.

**10.1.1.7 Stirrer** — mechanical.

## 10.1.2 Procedure

**10.1.2.1 Preparation of test specimen** — The material shall be prepared as follows by either of the methods:

- a) *Pour method* — Melt the sample just above its softening point. Stir thoroughly avoiding trapping of air bubbles. Place ring on a brass plate, amalgamated to prevent adherence of sample. Pour enough sample into ring to provide an excess when cool. After cooling for about 30 minutes, remove excess sample with knife, slightly heated. Proceed with test as in 10.1.2.2 within 4 hours.
- b) *Powder method* — Break sample into pieces not larger than 3.0 mm. Mix thoroughly and quarter down to a portion of 50 to 75 g. Grind to powder and separate the portion passing through 300-micron IS Sieve and retained on 75-micron IS Sieve. Assemble mortar and add powdered resin to about half-full sieve. Insert pestle and compact powder by striking pestle with a mallet. Remove ring and scrape off excess resin from top. If top and bottom surfaces are not smooth and flush with ring, discard sample and repeat preparation with a clean ring and fresh powder.

**10.1.2.2** Two procedures are prescribed depending upon the softening point of the resin. Tests shall be carried out in duplicate.

- a) *For materials of softening point above  $80^{\circ}\text{C}$*  — Assemble the apparatus and fill the bath to a height of 5 cm above the upper surface of the rings with glycerine at a temperature of  $35^{\circ}\text{C}$ . Maintain the bath at  $35^{\circ}\text{C}$  for 15 minutes, after which place a ball previously raised to  $35^{\circ}\text{C}$ , in each ball guide by means of forceps. Apply heat to the bath and stir the liquid so that the temperature rises at uniform rate of  $5 \pm 0.5^{\circ}\text{C}$  per minute until the material softens and allows the ball to pass through the ring. The rate of temperature-rise shall not be averaged over the period of test and any test in which the rate of temperature-rise does not fall within the specified limits after the first three

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\*Specification for general purpose glass thermometers : Part 1 Solid stem thermometers ( second revision ).

minutes, shall be rejected. Record, for each ring and ball, the temperature shown by the thermometer at the instant the material surrounding the ball touches the bottom plate of the support or the bottom of the bath. Repeat the test if the difference between duplicate determinations exceeds 1°C.

- b) *For materials of softening point below 80°C* — Follow the procedure prescribed in 10.1.2.2 (a) except that freshly boiled distilled water is used in the bath in place of glycerine and starting temperature of test is 5°C instead of 35°C.

**10.1.3 Report** — Report, to the nearest 0.5°C, the mean of the temperatures recorded in duplicate determinations as softening point.

## 10.2 Capillary Tube Method

**10.2.0 General** — The temperature at which visually the change in state of the test specimen of resin in a capillary tube, enclosed in a heating chamber, is observed.

### 10.2.1 Materials

**10.2.1.1 Bismuth** — Analytical quality, melting point 271.3°C.

**10.2.1.2 Tin** — Analytical quality, melting point 231.9°C.

**10.2.1.3 Suitable pure compounds** — of known melting point.

### 10.2.2 Apparatus

**10.2.2.1 Melting apparatus** consisting of the following items ( see Fig. 11 ):

- a) *Cylindrical metal block* — The upper part of which is hollow and forms a chamber.
- b) *Metal plug* — With two or more holes, allowing a thermometer and one or more capillary tubes to be mounted into the metal block.
- c) *Heating system for the metal block* — Provided, for example, by an electrical resistance enclosed in the block.
- d) *Rheostat* — For regulation of the power input, if electrical heating is used.
- e) *Four windows of heat-resistant glass* — On the lateral walls of the chamber, diametrically disposed at right angles to each other. In front of one of these windows is mounted an eye-piece for observing the capillary tube. The other three windows are used for illuminating the inside of the enclosure by means of lamps.

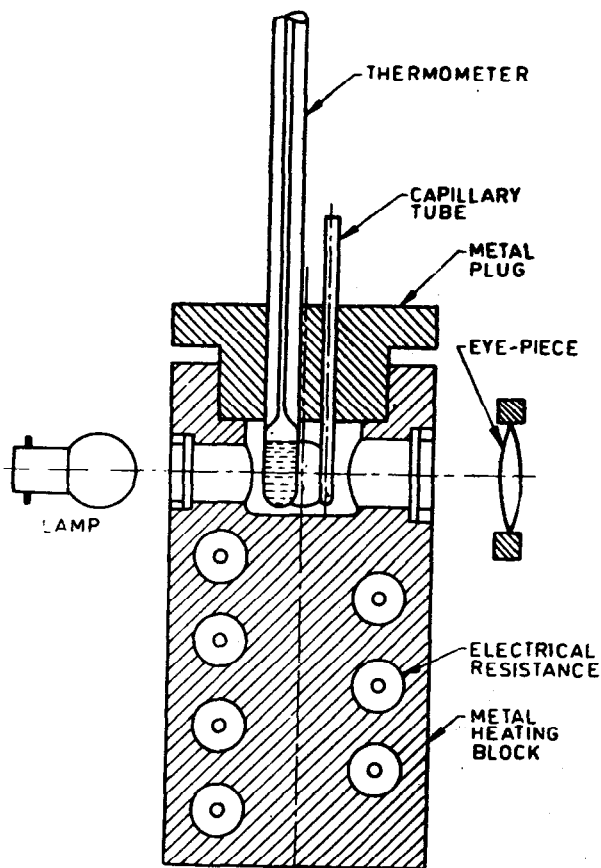


FIG. 11 MELTING APPARATUS WITH CAPILLARY TUBE

**10.2.3 Capillary Tube** — Of heat-resistant glass, closed at one end, and of maximum external diameter 2 mm.

**10.2.3.1 Precision thermometer** — Graduated from 20 to 300°C, in 1°C intervals.

#### 10.2.4 Test Specimen

**10.2.4.1** The test specimen shall be in the form of fine powder.

### 10.2.5 Procedure

**10.2.5.1** Before using the apparatus for the first time, and whenever the thermometer is changed, calibrate the apparatus following the procedure given below, but using, instead of specimen, one or more of the appropriate standards from 10.2.1 melting close to or covering the range of the melting points to be measured. Insert the thermometer in position in the apparatus. Insert the capillary tube containing the test specimen in the apparatus and heat with maximum power. When the temperature is about  $10^{\circ}\text{C}$  below the expected melting point, reset the rheostat to reduce the rate of temperature rise to  $2 \pm 0.5^{\circ}\text{C}$  per minute. Switch on the illuminating lamps of the apparatus. Observe the specimen and record the temperature indicated by the thermometer, at which the specimen melts. The melting point corresponds to that temperature at which the sharp edges of the specimen disappear. Repeat the operations described above using a second test specimen. If there is a difference of more than  $5^{\circ}\text{C}$  between the two determinations these results shall be rejected and two additional specimens tested.

### 10.2.6 Report

**10.2.6.1** Report to the nearest  $0.5^{\circ}\text{C}$ , or the mean, if the temperatures are recorded in duplicate, as melting point by capillary tube method.

### 10.2.7 Test Report

**10.2.7.1** The test report shall include the following particulars:

- a) Complete identification of the sample, that is, type, source of supply, manufacturer's code number, trade name, and any other information which is considered necessary;
- b) Method of test used;
- c) Melting point, in accordance with 10.2.5;
- d) Observations of any circumstances which may have affected the result; and
- e) Date of test.

## 11. MOISTURE CONTENT

**11.0 General** — Three methods are prescribed for determination of moisture content. In case of dispute Karl Fischer method shall be applicable.

### 11.1 Method 1 — Desiccator Method

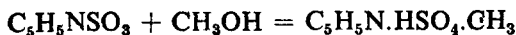
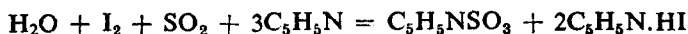
**11.1.1 Procedure** — Weigh accurately about 5 g of the material in a tared flat-bottomed porcelain dish and spread the material at the bottom

of the dish so as to form a thin layer of uniform thickness. Place the dish in vacuum over sulphuric acid in a desiccator at room temperature and dry to constant mass.

11.1.2 Calculate and express loss in mass as percentage of the mass of the material taken for test.

## 11.2 Method 2 — Karl Fischer Method

11.2.0 *Outline of the Method* — Karl Fischer method is based on the reaction between iodine and sulphur dioxide in the presence of water. Methanol and pyridine, besides acting as solvent and buffer, also take part in the reaction as follows:



### 11.2.1 Apparatus

11.2.1.0 *General* — All apparatus used should be dried before use. This may be done by rinsing it with dry acetone and then blowing hot air into it.

11.2.1.1 *Titration flask* — with a capacity of 60 to 75 ml, fitted with a rubber stopper with accommodation for the burette tip, a vent tube provided with a desiccant, and two platinum electrodes.

11.2.1.2 *Stirrer* — magnetic or propeller type.

11.2.1.3 *Potentiometer* — of resistance 2 000 ohms. Two bright platinum electrodes which are externally polarized by means of a 1.5-volt dry cell shall be connected through the potentiometer.

### 11.2.1.4 Microammeter

### 11.2.2 Reagents

11.2.2.1 *Methanol, dry* — To about 2 litres of methanol contained in a 4-litre round-bottom flask, add 0.5 kg of freshly burnt quick lime and fit a double-surface condenser carrying a guard tube containing anhydrous calcium chloride. Reflux gently on a water-bath for 6 hours, allow to stand overnight and then distil on a steam-bath, the receiver being protected from atmospheric moisture by a guard tube. Place a plug of glass wool at the mouth of the distillation head to retain any finely divided lime which tends to pass over with methanol. Discard the first 10 to 15 ml of the distillate and collect the methanol boiling at 65°C at 760 mmHg pressure.



**11.2.2.2 Pyridine, dry** — Reflux pyridine over sodium hydroxide pellets in a round-bottomed flask and distil the pyridine, collecting the fraction boiling between 114 and 116°C at 760 mm pressure, the receiver being protected from atmospheric moisture by a guard tube.

### 11.2.2.3 Fischer reagent

- a) **Solution A** — Dissolve 125 g of iodine in 650 ml of methanol contained in a flask; add into the flask 200 ml of pyridine and immediately close the flask tightly.
- b) **Solution B** — Measure 100 ml of pyridine into a dry flask fitted with a two-hole rubber stopper with an inlet tube reaching nearly to the bottom of the flask and a short outlet tube protected by a guard tube. Cool the flask on an ice-bath. Connect the inlet tube to a source of dry sulphur dioxide gas and pass the gas into pyridine with continuous agitation until the volume of the liquid reaches 200 ml.
- c) Slowly add solution B to the cooled solution A, stopper immediately and shake well until the iodine is dissolved. Transfer the solution to an automatic pipette protected from absorption of moisture by a drying agent, and allow to stand for 24 hours before standardizing. The reagent deteriorates continuously and it should be standardized within one hour before use.

**NOTE** — Fischer reagent is extremely hygroscopic and precautions shall be taken to exclude all atmospheric moisture during the preparation, storage and use of the reagent.

**11.2.2.4 Standard water solution** — Weigh accurately about 2 g of water into a thoroughly dry 1-litre volumetric flask and dilute to mark with methanol. Retain sufficient quantity of the same methanol for a blank determination. Keep the solution in tightly closed containers.

### 11.2.3 Procedure

**11.2.3.0 General** — It is important that in carrying out the titration, care shall be taken to avoid contamination by atmospheric moisture at all stages.

**11.2.3.1 Determination of end point in Karl Fischer titration** — In many cases, the end point can be detected visually by the change, from a light brownish-yellow to an amber colour. But when the end point is not clearly defined, the electrometric method for determining the end point is adopted. The potentiometer is adjusted so that when a small excess (0.02 ml) of the reagent is present, a current of 50 to 150  $\mu$ A is recorded. The solution is continuously and efficiently stirred. At the beginning of the titration, a current of only a few microamperes will flow. After each

addition of reagent, the pointer of the microammeter is deflected but rapidly returns to the original position. At the end point, a deflection is obtained which endures for at least 30 seconds.

**11.2.3.2 Standardization of the Fischer reagent** — Pipette exactly 10 ml of methanol into a dry titration flask and titrate with the Fischer reagent to the end point (  $V_1$  ml ). Then pipette exactly 10 ml of standard water solution into the flask and titrate again to the end point. Note the total volume of Fischer reagent used (  $V_2$  ml ).

**11.2.3.3 Titration of the material** — Transfer 25 ml of methanol to the titration flask and titrate to the end point with the Fischer reagent. Do not record the volume consumed. Quickly transfer to the titrated liquid an accurately weighed quantity of the material containing 10 to 50 mg of water, stir vigorously and titrate to end point. Note the total volume of Fischer reagent used (  $V_3$  ml ).

#### 11.2.4 Calculation

$$\text{Water, percent by mass} = \frac{M_1 (V_3 - 2.5 V_1)}{10 M_2 (V_2 - V_1)}$$

where

$M_1$  = mass in mg of water contained in 10 ml of standard water solution,

$V_3$  = total volume in ml of Fisher reagent used in titration in 11.2.3.3,

$V_1$  = volume in ml of Fischer reagent used in titration of methanol in 11.2.3.2,

$M_2$  = mass in g of the material taken for test in 11.2.3.3, and

$V_2$  = total volume in ml of Fischer reagent used in titration in 11.2.3.2.

### 11.3 Determination of Free Water

**11.3.0 Outline of the Method** — The material is heated under reflux with an organic solvent which is immiscible with water. The carrier liquid distils into a graduated receiver carrying with it water which then separates to form the lower layer, the excess carrier liquid overflowing from the trap and returning to the still.

**11.3.1 Apparatus** — The Dean and Stark apparatus used for determination of water content has the following essential features.

**11.3.1.1 Flask** — of 500 ml capacity, as shown in Fig. 12, and made of hard resistance glass, well-annealed and as free as possible from striae and similar defects. Alternatively, a metal flask may be used.

**11.3.1.2 Condenser** — made of hard resistant glass, well-annealed and as free as possible from striae and similar defects, with shape and dimensions as shown in Fig. 13.

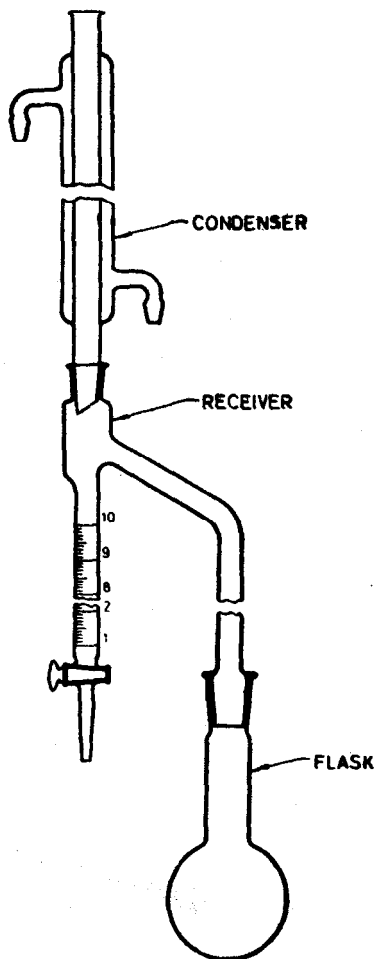
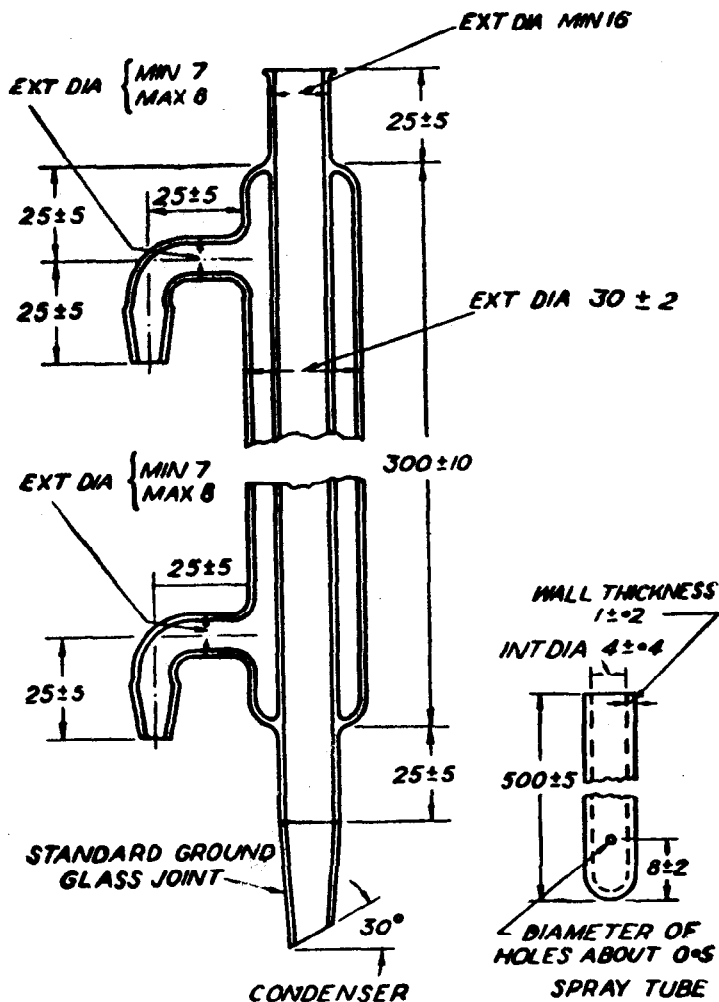


FIG. 12 DEAN AND STARK ASSEMBLY ( WITH 10-ml RECEIVER )



All dimensions in millimetres.

FIG. 13 CONDENSER AND SPRAY TUBE ( DEAN AND STARK APPARATUS )

**11.3.1.3 Spray tube** — made of glass, sealed at one end, having four small holes equidistantly placed around the wall near the closed end of the tube, with the shape and dimensions as shown in Fig. 13.

**11.3.1.4 Two-millilitre receiver** — made of hard resistant glass, well-annealed and as free as possible from striae and similar defects, provided with ground-glass joints, and of shape and dimensions given in Fig. 14. It consists essentially of the upper chamber together with the tube and ground joint leading to the flask and the graduated tube. When a metal flask is used, care shall be taken to provide an air-tight connection between the flask and the receiver. The graduated portion shall have a capacity of 2 ml at 20°C when filled to the highest graduation mark. The scale shall cover the range of 0.1 to 2 ml and shall be divided into intervals of 0.05 ml. The graduation marks corresponding to 0.5, 1.0, and 2.0 ml shall be numbered. The numbered graduation marks shall be carried completely round the tube. The graduation marks corresponding to 0.15, 0.25, 0.35 ml and so on up to and including 1.95 ml, shall be carried half-way round the tube. The remaining graduation marks shall be intermediate in length and shall project equally at each end beyond the shortest graduation marks. The error at any point on the scale shall not exceed  $\pm 0.03$  ml and the difference between the errors at any two points shall not exceed 0.03 ml.

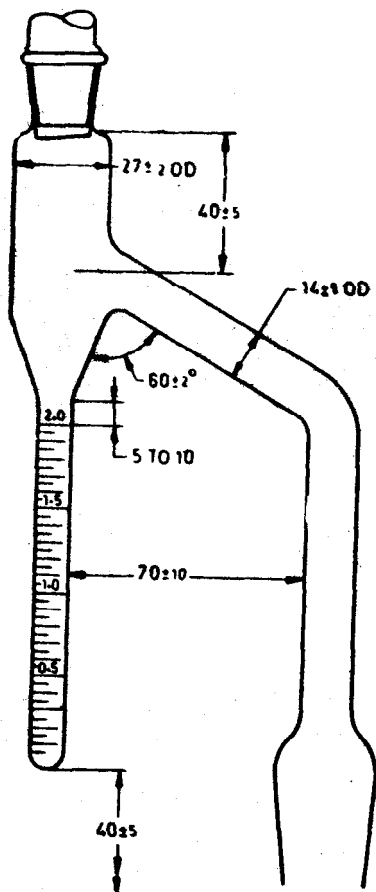
**11.3.1.5 Graduated cylinder** — 100 ml.

**11.3.2 Procedure** — Weigh 100 g of the material in the flask, add 100 ml of dry petroleum hydrocarbon solvent (boiling point 75 to 85°C) and 1 ml of dry ethyl acetate (conforming to IS : 229 - 1972\*) or amyl acetate (conforming to IS : 231 - 1957†) and thoroughly mix the contents to the flask. Pour petroleum hydrocarbon solvent into the receiver up to the level of the side tube. Attach the flask to the Dean and Stark condensing and collecting system and heat the flask at such a rate that the condensate falls from the end of the condenser at a rate of two to five drops per second. Continue the distillation until condensed water is no longer visible in any part of the apparatus except at the bottom of the graduated tube and until the volume of water collected remains constant. Remove the persistent ring of condensed water in the condenser tube, if any, by increasing the rate of distillation by a few drops per second. Wash droplets of water which adhere to the lower end of the condenser tube into the receiver with petroleum hydrocarbon solvent, using the spray tube.

**11.3.2.1** Note the volume in millilitres of water in the receiver at the temperature at which the sample was measured. Assuming the density of

\*Specification for ethyl acetate (second revision).

†Specification for amyl acetate.



All dimensions in millimetres.

FIG. 14 2-ml RECEIVER

1.00 g/ml for the water collected in the receiver, calculate the percentage of water ( by mass ) in the material.

## 12. VOLATILE AND NON-VOLATILE MATTER

### 12.1 Apparatus

**12.1.1 Flat-Bottomed Dish** — of glass, tinplate or aluminium, approximately 75 mm in diameter.

**12.1.2 Thin Glass Rod or Metallic Wire**

**12.1.3 Air Oven** — capable of maintaining the specified temperature.

**12.2 Procedure**

**12.2.1 Test Portion** — Dry the glass, tinplate or aluminium dish and the glass rod or wire in the oven at the test temperature ( *see* Table 6 ) and allow to cool at room temperature in a desiccator. Weigh the dish containing the glass rod or wire, to the nearest milligram and then weigh into the dish, to the same accuracy, approximately  $2 \pm 0.2$  g of the product under test as rapidly as possible to avoid loss by evaporation, making sure that it is evenly distributed over the surface of the dish. If the product contains a highly volatile solvent, or in the case of a reference test, weigh it by difference from a weighing bottle or from a suitable syringe into the dish. In the case of products which are very viscous or which form skins, distribute the test portion uniformly with the glass rod or wire, if necessary, after addition of 2 ml of an appropriate solvent.

**12.2.2 Determination** — Place the dish with the rod or wire and the test portion in the air oven previously adjusted at the agreed or specified temperature. Leave it in the oven at this temperature for the agreed or specified period ( *see* Table 6 ).

---

**TABLE 6 RECOMMENDED TEST CONDITIONS FOR VARIOUS RESIN SOLUTIONS**

Sl. No.	MEDIUM	TEMPERATURE	DRYING TIME
(1)	(2)	°C (3)	h (4)
i) Alkyd and epoxide ester resins		125	1
ii) Non-modified epoxide resins		140	3
iii) Phenolic resins		135	1
iv) Acrylic resins		} By agreement between the interested parties	
v) Amino resins			
vi) Bituminous products			
vii) Polyamides resins			

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To break up any surface skin, remove the dish together with the rod or wire from the oven after a short period of heating and stir the material with the rod or wire. Replace the dish and rod or wire in the oven.

NOTE — In the absence of an agreement or a specification concerning the temperature or duration of heating, the test conditions should be  $105 \pm 20^\circ\text{C}$  for 3 hours.

**12.2.3** When the agreed or specified period of heating is completed, transfer the dish and rod or wire to a desiccator, allow to cool to room temperature and re-weigh to the nearest milligram.

**12.2.4** Perform at least two determinations on the same prepared sample.

**12.3 Calculation** — Calculate the content of volatile matter or of non-volatile matter as a percentage by mass of the product tested, by the following formulae:

$$V = 100 \frac{(m_1 - m_2)}{m_1}$$

$$NV = 100 \frac{m_2}{m_1}$$

where

$V$  = volatile matter, as a percentage by mass;

$NV$  = non-volatile matter, as a percentage by mass;

$m_1$  = mass, in milligrams, of the test portion before heating;

$m_2$  = mass, in milligrams, of the test portion before heating under the specified conditions.

Report the result in percent by mass to the first decimal place, as the arithmetic mean of the determinations performed.

### 13. MATTER INSOLUBLE IN TOLUENE ( OR BENZENE )

**13.1 Procedure** — Dissolve about 10 g of the powdered material, accurately weighed, in 50 ml of toluene ( or benzene ) kept on the boil under a reflux condenser. When the material has completely dissolved, filter the solution through either counterpoised, good quality, grease-free filter paper or a weighed Gooch crucible provided with asbestos mat or a sintered filter and dried at  $100 \pm 2^\circ\text{C}$ . If filter papers are used, heat them to a temperature of  $100 \pm 2^\circ\text{C}$  and reduce to equal mass by removing the apex of the heavier paper. When filtering, place the uncut paper inside the originally heavier paper so that the residue is retained by the inner paper,



but both of them are equally subjected to any action exerted by the solvent. Transfer any insoluble residue left on the filter paper or the Gooch crucible or the sintered filters with the aid of the warm solvent and wash the contents with a minimum quantity of the warm solvent until a few drops of the filtrate yield no residue on evaporation. Dry the residue in an air-oven at a temperature of  $100 \pm 2^\circ\text{C}$  for one hour, and weigh.

### 13.2 Calculation

Matter insoluble in toluene

$$(\text{ or benzene }), \text{ percent by mass} = \frac{M_1}{M_2} \times 100$$

where

$M_1$  = mass in g of the residue, and

$M_2$  = mass in g of the material taken for the test.

## 14. ASH CONTENT

### 14.1 Procedure

**14.1.1** Weigh accurately about 5 g of the material in a tared crucible and ignite over a Bunsen burner till the resin is mostly burnt off and then heat the dish in a muffle furnace kept at a temperature of  $700 \pm 50^\circ\text{C}$  to constant mass.

**14.1.2** Calculate the amount of ash so obtained as percentage of the mass of the material taken for test.

## 15. DETERMINATION OF ACID VALUE

**15.0 Outline of the Method** — Acid value is determined by finding the number of milligrams of potassium hydroxide required to neutralize free fatty acids contained in one gram of the material.

### 15.1 Reagents

**15.1.1 Ethanol-Benzene Mixture** — Mix equal volumes of ethanol (95 percent) and benzene and neutralize the mixture with ethanolic potassium hydroxide solution (0.1 N) in the presence of phenolphthalein as indicator.

**15.1.2 Standard Alcoholic Potassium Hydroxide Solution** — 0.01 N.

**15.1.3 Phenolphthalein Indicator Solution** — Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent rectified spirit ( conforming to IS : 323-1959\* ).

**15.2 Procedure** — Weigh accurately, to the nearest 0.01 g, about 10 g, depending upon the free acids, of the material into a conical flask. Add 150 ml of ethanol-benzene mixture to the material, shake and warm, if necessary, to dissolve. Titrate the solution with standard alcoholic potassium hydroxide solution using phenolphthalein as indicator.

### 15.3 Calculation

$$\text{Acid value} = 56.1 \frac{VN}{M}$$

where

$V$  = volume in ml of standard alcoholic potassium hydroxide solution used,

$N$  = normality of standard potassium hydroxide solution, and

$M$  = mass in g of the material taken for the test.

## 16. DETERMINATION OF IODINE VALUE ( WIJS )

**16.0 General** — The material is treated, in carbon tetrachloride medium, with a known excess of iodine monochloride solution in glacial acetic acid ( Wijs solution ). The excess of iodine monochloride is treated with potassium iodide and the liberated iodine estimated by titration with sodium thiosulphate solution.

### 16.1 Reagents

**16.1.1 Potassium Bichromate** — conforming to IS : 250-1964†.

**16.1.2 Concentrated Hydrochloric Acid** — conforming to IS : 265-1976‡.

**16.1.3 Potassium Iodide Solution** — Prepare a fresh solution by dissolving 10 g of potassium iodide, free from potassium iodate, in 90 ml of water.

**16.1.4 Starch Solution** — Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into one litre of boiling water. Boil for three minutes. Allow to cool and decant off the supernatant clear liquid.

\*Specification for rectified spirit ( revised ).

†Specification for potassium bichromate, technical and analytical reagent ( revised ).

‡Specification for hydrochloric acid ( second revision ).

**16.1.5 Standard Sodium Thiosulphate Solution** — approximately 0.1 N. Dissolve approximately 24.8 g of sodium thiosulphate crystals (  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  ) in water which has been well boiled to free it from carbon dioxide and make up to 1 000 ml. Store the solution in a cool place in a dark-coloured stock bottle with a guard tube filled with soda lime. After storing the solution for about two weeks, filter, if necessary, and standardize it as prescribed under 16.1.5.1.

**16.1.5.1** Weigh accurately about 5.0 g of finely ground potassium dichromate which has been previously dried to a constant mass at  $105 \pm 2^\circ\text{C}$  into a clean one-litre volumetric flask. Dissolve in water, make up to the mark; shake thoroughly and keep the solution in a cool dark place. For standardization of sodium thiosulphate, pipette 25 ml of this solution into a clean glass stoppered 250-ml conical flask or bottle. Add 5 ml of concentrated hydrochloric acid and 15 ml of a 10-percent potassium iodide solution. Allow to stand in the dark for 5 minutes and titrate the mixture with the solution of sodium thiosulphate, using starch solution as an internal indicator towards the end. The end point is taken when the blue colour changes to green. Calculate the normality ( $N$ ) of the sodium thiosulphate solution as follows:

$$N = \frac{25 M}{49.03 V}$$

where

$M$  = mass in g of the potassium dichromate, and

$V$  = volume in ml of sodium thiosulphate solution required for the titration.

**16.1.6 Iodine Crystals** — re-sublimed.

**16.1.7 Acetic Acid** — glacial, 99 percent, having a melting point of  $14.8^\circ\text{C}$  and free from reducing impurities. Determine the melting point of the acetic acid and test it for reducing impurities as follows:

- a) **Determination of melting point** — Take a 15-cm long test-tube and fill it to about two-thirds with the acetic acid. Insert into the acid a thermometer satisfying the requirements specified under 16.2 through a cork stopper fitting the test-tube. The amount of acid should be at least double the quantity required to cover the bulb of the thermometer when the bottom of the latter is 12 mm from the bottom of the test-tube. Suspend this tube within a larger test-tube through a cork. Cool the acid by immersing the assembly in ice water until the temperature is  $10^\circ\text{C}$ , then withdraw the assembly from the ice water and stir the acid rather vigorously for a few moments, thus causing the super-cooled liquid to crystallize

partially. Take thermometer readings every 15 seconds and consider as the true melting point that temperature at which the reading remains constant for at least 2 minutes.

- b) *Test for reducing impurities ( potassium permanganate test )* — Dilute 2 ml of the acetic acid with 10 ml of water and add 2 drops of 0.1 N potassium permanganate solution and maintain at  $27 \pm 2^\circ\text{C}$ . The test shall be taken as having been satisfied if the pink colour is not discharged at the end of two hours.

**16.1.8 Chlorine Gas** — dry.

**16.1.9 Iodine Trichloride** (  $\text{ICl}_3$  )

**16.1.10 Iodine Monochloride** (  $\text{ICl}$  ) — 98 percent chemically pure.

**16.1.11 Wijs Iodine Monochloride Solution** — Prepare this solution by one of the following three methods, and store in a glass-stoppered bottle in a cool place, protected from light:

- a) Dissolve 13 g of iodine in one litre of acetic acid, using gentle heat if necessary, and determine the strength by titration with standard sodium thiosulphate solution. Set aside 50 to 100 ml of the solution and introduce chlorine gas into the remainder until the characteristic colour change occurs and the halogen content is nearly doubled as ascertained again by titration. If the halogen content has been more than doubled, reduce it by adding the requisite quantity of the iodine-acetic acid solution. A slight excess of iodine does no harm, but avoid an excess of chlorine.

*Example:*

If the titration of 20 ml of original iodine-acetic acid solution requires 22 ml of standard sodium thiosulphate, 20 ml of the finished Wijs solution should require between 43 and 44 ml (and not more than 44 ml) of the same sodium thiosulphate solution.

- b) Dissolve 8 g of iodine trichloride in approximately 450 ml of acetic acid. Dissolve separately 9 g of iodine in 450 ml of acetic acid, using heat, if necessary. Add gradually the iodine solution of the iodine trichloride until the colour has changed to reddish-brown. Add 50 ml more of iodine solution and dilute the mixture with acetic acid till 10 ml of the mixture is equivalent to 20 ml of standard thiosulphate solution when the halogen content is estimated by titration in the presence of an excess of potassium iodide and water. Heat the solution to  $100^\circ\text{C}$  for 20 minutes, and cool. Prevent access of water vapour in preparing the solution.

- c) Dissolve 10 ml of iodine monochloride in about 1 800 ml of glacial acetic acid (chemically pure) and shake vigorously. Pipette 5 ml of this, add 10 ml of potassium iodide solution and titrate with 0.1 N standard sodium thiosulphate solution, using starch solution as indicator. Adjust the volume of the solution till it is approximately 0.2 N.

**16.1.12 Carbon Tetrachloride or Chloroform** — inert to Wijs solution.

## 16.2 Apparatus

**16.2.1 Thermometer** — with the following characteristics:

- a) *Type* — etched/marked stem glass.
- b) *Liquid* — mercury.
- c) *Filling above liquid* — evacuated or nitrogen gas.
- d) *Temperature range* — minus 2 to 68°C.
- e) *Subdivisions* — 0.2°C.
- f) *Total length* — 385 to 390 mm.
- g) *Stem diameter* — 6 to 7 mm.
- h) *Stem construction* — plain or lens front. The cross-section of the lens front type shall be such that it will pass through an 8-mm ring gauge but will not enter a 5-mm slot gauge.
- j) *Bulb diameter* — from 5.5 mm to not greater than the diameter of the stem
- k) *Bulb length* — 15 to 25 mm.
- m) *Distance from bottom of bulb to minus 2°C mark* — 50 to 60 mm.
- n) *Distance from 68°C mark to top of thermometer* — 20 to 35 mm.
- p) *Length of unchanged capillary between the highest graduation mark and the expansion chamber* — 10 mm.
- q) *Expansion chamber* — to permit heating to at least 85°C.
- r) *Top finish* — glass ring.
- s) *Longer graduation lines* — at each 1°C mark.
- t) *Graduation numbered* — at zero and at each multiple of 2°C.
- u) *Immersion* — 45 mm. A line shall be etched around the stem, 45 mm from the bottom of the bulb.

w) *Maximum scale error permitted at any point* —  $0.2^{\circ}\text{C}$ .

y) *Standardization* — The thermometer shall be standardized at the melting point of ice and at intervals of approximately  $20^{\circ}\text{C}$ , for the condition of 45 mm immersion and for an average stem temperature of the emergent mercury column of  $25^{\circ}\text{C}$ .

**16.3 Procedure** — Melt the sample, if it is not already completely liquid, and filter through a filter paper to remove any impurities and the last traces of moisture. Make sure that the sample as well as the glass apparatus used is absolutely clean and dry. Weigh accurately 0.15 to 0.20 g of the material into a clean dry 500-ml iodine flask or well ground glass-stoppered bottle to which 25 ml of carbon tetrachloride have been added, and agitate to dissolve the contents. The mass of the sample shall be such that there is an excess of 50 to 60 percent of Wijs solution over that actually needed. Add 25 ml of the Wijs solution and replace the glass stopper after wetting with potassium iodide solution; swirl for intimate mixing, and allow to stand in the dark for  $1\frac{1}{2}$  h at  $27 \pm 2^{\circ}\text{C}$ . Carry out a blank test simultaneously under similar experimental conditions. After standing, add 15 ml of potassium iodide solution and 150 ml of water, rinsing in the stopper also, and titrate the liberated iodine with standard sodium thiosulphate solution, swirling the contents of the bottle continuously to avoid any local excess until the colour of the solution is straw yellow. Add one millilitre of the starch solution and continue the titration until the blue colour formed disappears after thorough shaking with the stopper on.

#### 16.4 Calculation

$$\text{Iodine value} = \frac{12.69 (B-S) N}{M}$$

where

$B$  = volume in ml of standard sodium thiosulphate solution required for the blank,

$S$  = volume in ml of standard sodium thiosulphate solution required for the sample,

$N$  = normality of the standard sodium thiosulphate solution, and

$M$  = mass in g of the material taken for the test.

#### 17. DETERMINATION OF FREE HYDROXYL VALUE (ACETYL VALUE)

**17.0 Outline of the Method**— The material is acetylated with acetic anhydride and pyridine. The excess anhydride is hydrolyzed and titrated against standard ethanolic potassium hydroxide.

**17.1 Reagents**

**17.1.1 Acetic Anhydride-Pyridine Reagent** — Mix 7 parts of pure anhydride with 3 parts of pure, freshly distilled pyridine.

**17.1.2 *n*-Butanol** — conforming to IS : 361-1973\*.

**17.1.3 Mixed Indicator Solution** — Mix 1 part of 0.1 percent aqueous neutralized cresol red solution and 3 parts of 0.1 percent aqueous thymol blue solution.

**17.1.4 Standard Ethanolic Potassium Hydroxide Solution** — 0.5 N.

**17.2 Procedure** — Weigh accurately about 2 g of the material into a 250 ml iodine flask and add 5 ml of the acetic anhydride-pyridine reagent. Insert the glass stopper previously lubricated with a little pyridine, and heat the flask on a water-bath for 45 to 60 minutes. After refluxing, add 10 ml of water and hydrolyze excess acetylating agent. The addition of water is done by placing it in the stopper-well of the flask, introduced by easing up the stopper, contents mixed thoroughly and left for 2 minutes on the water-bath. Cool the flask to room temperature under tap, rinse the stopper with *n*-butanol and catch the washings in the flask. Add two or three drops of the mixed indicator and titrate against 0.5 N standard ethanolic potassium hydroxide solution. Carry out a blank simultaneously using all the reagents, but without the sample.

**17.3 Calculation**

$$\text{Free hydroxyl value, mg of potassium hydroxide per g of material} = \frac{(V_1 - V_2) \times N \times 56.1}{M}$$

where

$V_1$  = volume in ml of standard ethanolic potassium hydroxide solution used in the blank,

$V_2$  = volume in ml of standard ethanolic potassium hydroxide solution used in the test,

$N$  = normality of standard ethanolic potassium hydroxide solution, and

$M$  = mass in g of the material taken for test.

**18. FLASH POINT ( ABEL )**

**18.0 Outline of the Method** — The sample suitably cooled is placed in the cup of the Abel apparatus and heated at a prescribed rate. A small test

\*Specification for normal butyl alcohol, technical ( *second revision* ).

flame is directed into the cup at regular intervals and the flash point is taken as the lowest temperature at which application of the test flame causes the vapour above the sample to ignite with a distinct flash inside the cup.

**18.1 Apparatus** — The apparatus to be employed shall be the Abel petroleum testing apparatus or Abel apparatus modified by having an oil cup provided with a stirrer, constructed to the dimensions and tolerances prescribed in 18.1.1 to 18.1.3. The apparatus and thermometers shall bear a test certificate of the National Physical Laboratory, New Delhi, or any other institution authorized by the Government of India to issue such a certificate.

**18.1.1 Oil Cup** — The oil cup ( *A* in Fig. 15 ) is made of brass or gunmetal and consists of a cylindrical vessel open at the top and fitted on the outside with a flat circular flange projecting at right angles. Within the cup, fixed through the wall and silver-soldered or brazed in place, there is a gauge ( *B* in Fig. 15 ) consisting of a piece of wire bent upwards and terminating in a point. The dimension of the cup and gauge shall be as follows:

<i>Cup:</i>	<i>Dimension</i>
Wall and bottom thickness	1.422 mm ( 17 SWG )
Internal diameter	$50.80 \pm 1.27$ mm ( $2.00 \pm 0.05$ in )
Internal depth	$55.88 \pm 1.27$ mm ( $2.20 \pm 0.05$ in )
<i>Flange:</i>	
Thickness	1.422 mm ( 17 SWG )
Width	$12.70 \pm 1.27$ mm ( $0.15 \pm 0.05$ in )
Distance of upper side from top edge of cup	$9.52 \pm 1.27$ mm ( $0.375 \pm 0.05$ in )
<i>Gauge:</i>	
Thickness, <i>Min</i>	3.251 mm ( 10 SWG )
Distance of point from level of upper edge of cup	$17.78 \pm 0.13$ mm ( $0.700 \pm 0.005$ in )

**18.1.2 Cover of the Oil Cup** — The cup is provided with close fitting cover ( *C* in Fig. 15 and Fig. 16 ) of the same material of which the cup is made, with a downward projecting rim just reaching the flange on the cup. The requirements for the cover and its ancillaries are as follows:

- a) The downward projecting rim is made solid with the top, or silver-soldered or brazed in place. Upon the cover are mounted a



thermometer socket ( *D* in Fig. 15 and Fig. 16 ), trunnions to support an oil test lamp ( *E* in Fig. 16 ), a pair of guides ( *F* in Fig. 15 and Fig. 16 ) in which a slide moves, and a white bead ( *G* in Fig. 16 ). The top of the cover is pierced by three rectangular holes symmetrically placed on a diameter, one in the centre and the other two as close as practicable to the inner sides of the cover-rim and opposite each other. These three holes are covered or uncovered by means of a slide ( *H* in Fig. 16 ) moving in suitably disposed guides. The slide has two perforations, one corresponding in all particulars to the centre hole in the cover and the other to one of the holes at the side. The movement of the slide is restricted by suitable stops, and its length and the disposition of the holes are such that at the outer extremity of the movement of the slide, the holes in the cover are simultaneously just completely opened, and at the inner extremity of the movement of the slide, they are completely closed.

- b) The trunnions supporting the test lamp are fixed on the top of the guides and the lamp is mounted in the trunnions so that it is free to oscillate. The lamp is provided with a jet to contain a wick, and is so arranged that when the slide is moved so as to uncover the holes, the oscillating lamp is caught by a pin fixed in the slide and tilted over the central hole in such a way that the lower edge of the cover bisects the circle formed by the bore of the jet when in the lowest position. The flame then occupies a central position within the hole in both directions. A suitably mounted gas-jet may be substituted for the lamp. If an oil test lamp is being used, it shall be prepared by fitting it with a piece of flat-plaited candle-wick and filling it with colza or rape oil up to the lower edge of the opening of the spout or wick-tube. The lamp shall be trimmed so that, when lighted, it gives a flame of about 4 mm diameter, and this size of flame, which is represented by the projecting white bead on the cover of the oil cup, is readily maintained by simple manipulation from time to time with a small wire trimmer. A gas test flame may be employed, the size of the flame being adjusted to the size prescribed above.
- c) The thermometer socket is the form of a split tube, mounted on a diameter at right angles to the diameter through the centre of the holes, and fitted at such an angle as to bring the bulb of the thermometer, when in place, vertically below the centre of the cover at the correct distance from it.
- d) A white bead of ivory or other suitable material, the dimensions of which represent the size of test flame to be used, is mounted in a visible position on the cover.

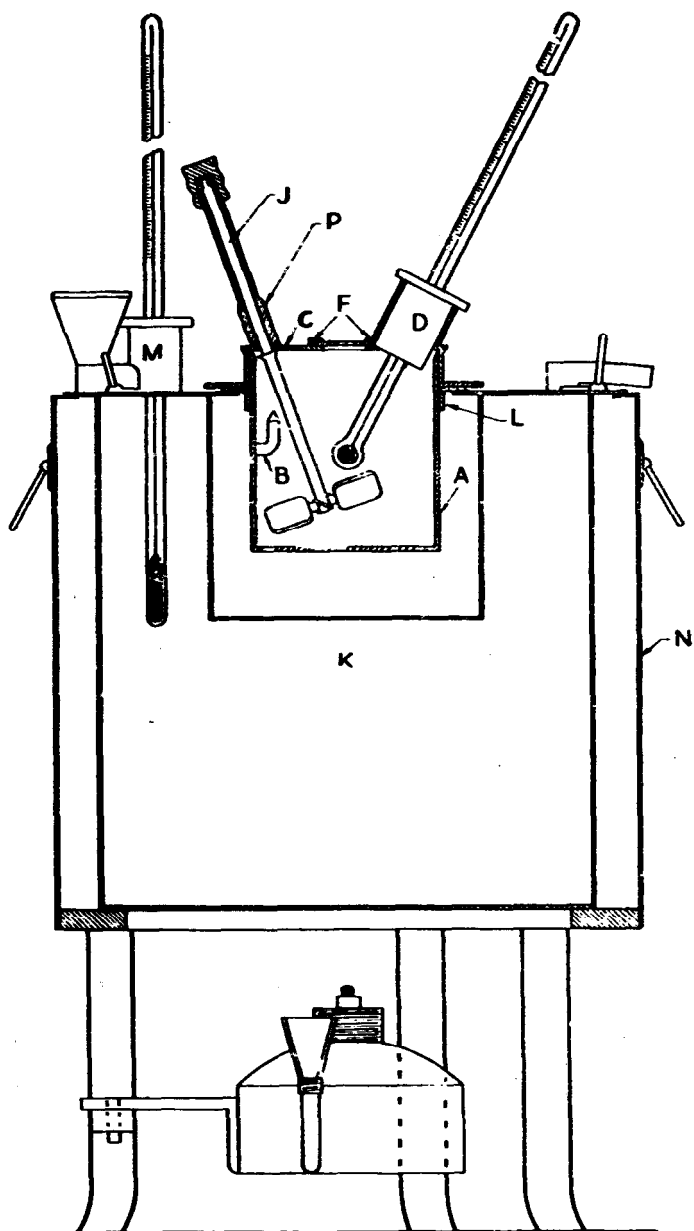


FIG. 15 ABEL FLASH POINT APPARATUS ( WITH STIRRER )

- e) The dimensions and tolerances of the cover, slide lamp, bead and thermometer socket shall be as follows:

<i>Cover:</i>	<i>Dimension</i>
Thickness	$1.27 \pm 0.38$ mm ( $0.50 \pm 0.015$ in )
Central hole, length ( in direction of slide )	$1.27 \pm 0.13$ mm ( $0.500 \pm 0.005$ in )
Central hole, width	$10.16 \pm 0.13$ mm ( $0.400 \pm 0.005$ in )
Peripheral holes, length ( in direction of slide )	$5.08 \pm 0.13$ mm ( $0.200 \pm 0.005$ in )
Peripheral holes, width	$7.62 \pm 0.13$ mm ( $0.300 \pm 0.005$ in )
<i>Slide:</i>	
Thickness	0.914 mm ( 20 SWG )
Width of upper surface	$12.7 \pm 0.2$ mm ( $0.5 \pm 0.01$ in )
<i>Lamp :</i>	
Overall length of jet	Approximately 15 mm (0.6 in) with a tolerance to suit the requirements for the position of the jet when tilted
Bore of jet at end	$1.58 \pm 0.13$ mm ( $0.0625 \pm 0.005$ in )
Bead, diameter	$3.8 \pm 0.2$ mm ( $0.15 \pm 0.01$ in )
Thermometer socket, internal diameter	$15.2 \pm 0.2$ mm ( $0.6 \pm 0.01$ in )
Length of short side measured from under surface of cover	Approximately 13 mm ( 0.5 in )
Length of long side measured from under surface of cover	Approximately 19 mm ( 0.75 in )
Distance of centre of socket from centre of cover measured on the under side	Approximately 18 mm ( 0.7 in )
Vertical depth of lowest part of thermometer below centre under side of cover ( subject to the correct placing of the thermometer when in position )	$38 \pm 2$ mm ( $1.5 \pm 0.1$ in )

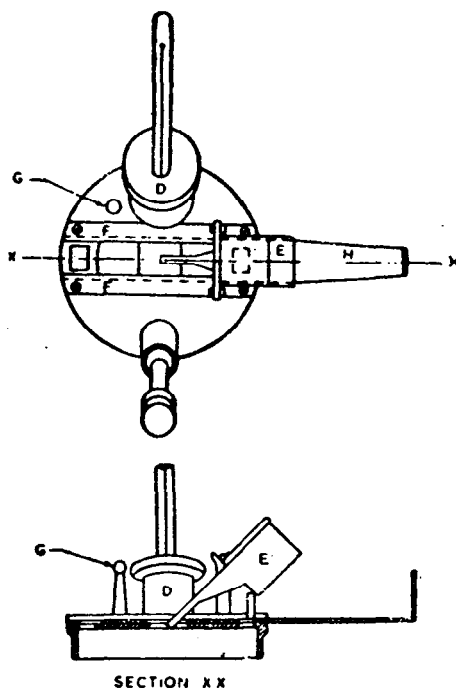


FIG. 16 COVER FOR ABEL FLASH POINT APPARATUS ( SLIDE OPEN )

**18.1.2.1 Device for the provision of the stirrer** — Provision may be made in the cover for the reception of a brass or gunmetal stirrer, which projects into the oil cup, for use with viscous materials only. The various components of this device are as follows:

- a) *Bush ( P in Fig. 15 )* — mounted on the cover in a position diametrically opposite the thermometer mounting, and its length is such and it is set at such an angle that the stirrer rod clears the oil-level gauge and the blades operate below the level of and without fouling the thermometer bulb. The bush is placed as near as practicable to the outer edge of the cover. A flat-headed cylindrical plug is provided for insertion in the bush when the stirrer is not in use.
- b) *Stirrer ( F in Fig. 15 )* — consists of a round stem having four blades or vanes silver-soldered in place, at one end. A collar is fixed on the stem so that when the stem is inserted into the bush

from below, it is arrested at a position such that the correct length protrudes into the oil cup. The top end of the stem is reduced and screwed.

- c) *Long sleeve* — having an internally-screwed knurled knob soldered to its upper end, passed over the upper end of the stem and screwed home. The length of the sleeve is such that a flat-faced collar at its lower end just comes into contact with the upper end of the bush, leaving the stirrer free to rotate without appreciable vertical play.

**18.1.2.2** The dimensions and tolerances of the stem, blades, sleeve, bore and collar are as follows:

<i>Stem:</i>	<i>Dimension</i>
Length, overall	$102 \pm 2$ mm ( or $4 \pm 0.1$ in )
Length, lower end to point of attachment of blades	Approximately 2 mm ( 0.1 in )
Length, lower end to upper surface of collar	$48 \pm 2$ mm ( $1.9 \pm 0.1$ in )
Length, upper surface of collar to lower end of thread	$51 \pm 2$ mm ( $2 \pm 0.1$ in )
Diameter	Approximately 3.5 mm ( 0.125 in )
Diameter of collar	Approximately 6 mm ( 0.25 in )
<i>Blades:</i>	
Thickness	1.422 mm ( 17 SWG )
Length, excluding root	$12.7 \pm 0.2$ mm ( $0.5 \pm 0.01$ in )
Breadth ( with all corners of blades rounded )	$7.9 \pm 0.2$ mm ( $0.3125 \pm 0.01$ in )
Angle	Approximately $45^\circ$
Sleeve, length	To suit stem giving free rotation with no appreciable vertical play when screwed home
Diameter of bore	Sliding fit on stem
Diameter of collar	Approximately 6 mm ( 0.25 in )

**18.1.3 Heating Vessels** — The heating vessel or bath ( *K* in Fig. 15 ) consists of two flat-bottomed cylindrical copper vessels placed coaxially, one inside the other, and soldered at their tops to a flat copper ring, greater in

outside diameter than the larger vessel and of smaller inside diameter than the smaller vessel. The space between the two vessels is thus totally enclosed and is used as a water-jacket.

#### 18.1.4 Other components of the apparatus are as follows:

- a) *Ring* — made of ebonite or fibre ( *L* in Fig. 15 ), of right angle section, is fitted into the hole in the centre of the flat ring forming the top of the bath and when the apparatus is in use, the oil cup fits into and its flange rests upon this ring so that the oil cup is centrally disposed within the heating vessel. The ring is secured in place by means of six small screws having their heads sunk below the surface of the ring, to avoid metallic contact between the bath and the oil cup.
- b) *Split socket* ( *M* in Fig. 15 ) — similar to that on the cover of the oil cup, but set vertically which allows a thermometer to be inserted into the water space. A funnel and overflow pipe also communicate with the water space through the top plate on which two loop handles are provided.
- c) *Cast iron tripod stand* — upon which the bath rests and to the ring of which is attached a cylindrical copper outer jacket ( *N* in Fig. 15 ), not less than 0.559 mm in thickness and  $165 \pm 2$  mm in diameter, flanged inwards at the top; and of such dimension that the bath, while resting firmly on the iron ring, just touches with its outward projecting flange the inward turned flange of the jacket. Two handles are provided on the outer jacket.

18.1.4.1 The dimensions and tolerances of the inner vessel, outer vessel, top plate and ring are as follows:

<i>Inner vessels:</i>	<i>Dimension</i>
Thickness	0.559 mm ( 24 SWG )
Internal diameter	$76.20 \pm 1.27$ mm ( $3.00 \pm 0.05$ in )
Internal depth	$63.5 \pm 1.27$ mm ( $2.50 \pm 0.05$ in )
<i>Outer vessel:</i>	
Thickness, <i>Min</i>	0.559 mm ( 24 SWG )
Internal diameter	$140 \pm 2$ mm ( $5.5 \pm 0.1$ in )
Internal depth	$146 \pm 2$ mm ( $5.75 \pm 0.1$ in )

*Top plate:*

Thickness, <i>Min</i>	0.914 mm ( 20 SWG )
Outer flange projection	$10 \pm 2$ mm ( $0.375 \pm 0.1$ in )
Diameter of central hole	To suit ebonite or fibre ring. Clearance not to exceed 2.5 mm ( 0.1 in )

*Dimension**Ebonite or fibre ring:*

Internal diameter	Easy fit on oil cup
External diameter flange	$69.8 \pm 0.6$ mm ( $2.75 \pm 0.02$ in )
Overall depth of spigot	$6.4 \pm 0.6$ mm ( $0.25 \pm 0.02$ in )
Thickness flange and spigot	$2.03 \pm 0.13$ mm ( $0.08 \pm 0.005$ in )

*Thermometer socket:*

Internal diameter	$15.2 \pm 0.2$ mm ( $0.60 \pm 0.1$ in )
Height from top of plate	$19.05 \pm 1.27$ mm ( $0.75 \pm 0.05$ in )

18.1.5 *Spirit Lamp* — for raising the temperature of the water-bath.

18.1.6 *Thermometers* — two, one for the oil cup and another for the water bath, conforming to the following specifications:

	<i>Oil Cup</i>	<i>Water Bath</i>
Range	10 to 65°C	32 to 88°C
Graduation	0.5°C	0.5°C
Longer lines at each	1°C and 5°C	1°C and 5°C
Figured at each	5°C	5°C
Overall length	$228 \pm 10$ mm ( 9 in approximately )	$228 \pm 10$ mm ( 9 in approximately )
Length of graduated portion, <i>Min</i>	120.6 mm ( 4.75 in )	90 mm ( 3.55 in )
Bulb, shape	Spherical	Cylindrical
Bulb, length	—	20 mm approximately ( 0.8 in approximately )

	<i>Oil Cup</i>	<i>Water Bath</i>
Bulb, diameter	$8.9 \pm 1.3$ mm ( 0.35 to 0.5 in )	Not greater than stem
Stem, diameter	6.1 to 7.1 mm ( 0.24 to 0.28 in )	6.1 to 7.1 mm ( 0.24 to 0.28 in )
Distance, bottom of bulb to lowest graduation mark	70 to 80 mm ( 2.75 to 3.15 in )	100 to 111 mm ( 3.95 to 4.35 in )
Top finish	Plain	Plain
Scale error not to exceed	$\pm 0.2^{\circ}\text{C}$	$\pm 0.5^{\circ}\text{C}$

An expansion chamber shall be provided. Swelling shall be provided in the stems to ensure that when the thermometers are fixed in their brass collars, the distance from the top of the collar to the bottom of the bulb is  $60.96 \pm 1.27$  mm (  $2.40 \pm 0.05$  in ) for the oil cup thermometer and  $89 \pm 2$  mm (  $3.5 \pm 0.1$  in ) for the water-bath thermometer. The brass collar shall be of the following dimensions:

Outside diameter	Push fit in socket
Thickness of tube	0.707 mm
Thickness of flange	$2.540 \pm 0.254$ mm ( $0.100 \pm 0.001$ in )

**18.1.7 Metronome** — To give 75 to 80 beats per minute. Alternatively a pendulum of 60 cm effective length may be used in place of the metronome, counting one beat from one extremity of the swing to the other.

## 18.2 Procedure

**18.2.1 Method A** — This method is applicable for liquids flashing between 19 and  $32^{\circ}\text{C}$  inclusive.

**18.2.1.1** Fill the water-bath to overflow with warm water, insert the water-bath thermometer and adjust the bath temperature to  $54^{\circ}\text{C}$  at the beginning of a test. Do not apply heat to the water-bath at any time during the test.

**18.2.1.2** Adjust the temperature of the sample to between 0 and  $10^{\circ}\text{C}$  ( sometimes there is a legal requirement that before a test is begun the material and oil cup, cover and thermometer be brought separately to a temperature of  $10^{\circ}\text{C}$ . ) Place the cup on a level surface in good light.



and fill it with sample until the liquid just reaches the point of the gauge fixed to the wall of the cup. Place the cover, with the slide closed, on the cup and press it down so that its edge rests on the rim of the cup. Place the cup in the water-bath (see Note), taking care to avoid wetting the sides of the cup above the liquid level. Insert the oil cup thermometer, seating the collar firmly in the cover.

NOTE—The cup shall be an easy fit.

**18.2.1.3** Light the test flame and adjust its size to approximately 4 mm diameter and maintain it at that size throughout the test, comparing it frequently with the projecting white bead mounted on the cover of the oil cup. Where an oil-fed flame is used, place the lamp in position on its trunnions on the cover.

**18.2.1.4** When the temperature of the oil reaches 18°C, apply the test flame by slowly opening the side in the cover while the metronome beats three times and closing it during the fourth beat.

**18.2.1.5** Apply the test flame in this manner after every 0.5°C rise in temperature until a distinct flash occurs in the interior of the cup or a temperature corresponding to a corrected flash point of 32°C is reached. Record the temperature of the sample when the flash occurs and also the barometric pressure in millimetres of mercury. The bluish halo, which sometimes surrounds the test flame, shall not be mistaken for the flash.

**18.2.2 Method B** — This method is applicable for liquids flashing between 33°C and 49°C inclusive.

**18.2.2.1** Proceed as prescribed in 18.2.1 but fill the air chamber which surrounds the cup with cold water to a depth of 38 mm and fill the water-bath with cold water. Heat the water-bath during the test so that the temperature of the sample is raised at the rate of 1 to 1.5°C per minute, and make the first application of the test flame when the temperature has reached 27°C.

**18.3 Barometric Pressure** — Observe and record the barometric pressure. Correct the observed flash point for air pressure using the formula:

$$t_n = t_b - \frac{b - 760}{30}$$

where

$t_n$  = flash point in °C at the standard pressure of 760 mm of mercury,

$t_b$  = flash point in °C at the observed pressure of  $b$  mm of mercury, and

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*b* = observed barometric pressure in mm of mercury.

**18.4 Precision** — Results of duplicate tests shall not differ by more than the following amounts:

*Flash Point*

5 to 65°C

*Repeatability*

± 0.8°C

*Reproducibility*

± 1.2°C

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**AMENDMENT NO. 1    JANUARY 1990**  
**TO**  
**IS : 354 ( Part 1 ) - 1987 METHODS OF SAMPLING**  
**AND TEST FOR RESINS FOR PAINTS**

**PART 1    GENERAL TEST METHODS**

*( Second Revision )*

*( Page 4, clause 0.2, line 7 )* — Delete the words 'chlorinated rubber'.

*( Page 4, clause 0.2, last line )* — Delete 'Part 8'.

( CDC 50 )

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**AMENDMENT NO. 2 JULY 2004**  
**TO**  
**IS 354 ( PART 1 ) : 1987 METHODS OF SAMPLING**  
**AND TEST FOR RESINS FOR PAINTS**

**PART 1 GENERAL TEST METHODS**

*( Second Revision )*

*( Page 8, clause 6.2.1.1 )* — Insert the following new clause after **6.2.1.1**:

**‘6.2.1.1.1 Calibration**

Gardner colour secondary standards shall be calibrated as per procedure described in Annex A.’

*( Page 14, clause 7.2.3 )* — Insert the following note after **7.2.3**:

“NOTE — For determination of relative density of viscous/semi-solid materials, mass per volume cup method as prescribed in IS 101 (Part 1/Sec 7) ‘Method of sampling and test for paints, varnishes and related products : Part 1 Test on liquid paints (general and physical), Section 7 Mass per 10 litres ( *third revision* )’ may also be used.”

*( Page 17, clause 8.2 )* — Substitute the method described in **3** of IS 101 (Part 1/Sec 5) ‘Methods of sampling and test for paints, varnishes and related products : Part 1 Test on liquid paints ( general and physical ), Section 5 Consistency ( *third revision* )’ for the existing method given in **8.2**.

*( Page 19, clause 8.3.1.1 )* — Insert the following new clause after **8.3.1.1**:

**‘8.3.1.1.1 Calibration**

Ostwald U-tube viscometer shall be calibrated as per procedure described in Annex B.’

*( Page 23, clause 8.4.1.1 )* — Insert the following new clause after **8.4.1.1**:

**‘8.4.1.1.1 Calibration**

The Brookfield viscometer shall be calibrated as per procedure described in Annex C.’

*( Page 62, clause 18.4 )* — Insert the following Annexes A, B and C at the end:

**ANNEX A**  
**(Clause 6.2.1.1.1)**

**A-1 PROCEDURE**

Prepare the Gardner colour reference standard solutions as described in 6.2.1.1. Fill the Gardner tubes with the solutions as per Table 2 and mark them with numbers 1 to 18. Compare visually each Gardner tube of test set with corresponding approved standard against white background provided in Gardner colour standard set. Comparison should be done for brightness and saturation of colour, ignore hue difference.

**A-2 ACCEPTANCE CRITERIA**

Gardner colour standards under test are stated to be calibrated when each standard under test matches closely with reference standard. In the event of a standard not matching, replace by the new.

**ANNEX B**  
**(Clause 8.3.1.1.1)**

**B-0 SCOPE**

This procedure describes the method for calibration of Ostwald U-tube viscometers, used for determining viscosity of liquids in the range of 1 cS to 20 000 cS.

**B-1 APPARATUS**

**B-1.1** Constant temperature water bath.

**B-1.2** Calibrated thermometer of range (0 to 100°C) and of 0.5°C least count.

**B-1.3** Analytical grade chemicals (such as ethylene glycol, nitrobenzene, mono chlorobenzene, xylene, toluene, butyl ethers of diethylene glycol or di-propylene glycol).

**B-1.4** Stopwatch of 1 s least count.

**B-2 PROCEDURE**

**B-2.1** Clean the U-tube viscometer, if necessary, by dipping in chromic acid. Rinse viscometer with water and acetone. Dry the viscometer by passing dry air through it.

**B-2.2** Suspend the clean viscometer with the help of a stand, in water bath maintained at a temperature at which viscosity of liquid is to be determined taking care to see that capillary arm is vertical. Fill the viscometer with the liquid of known viscosity up to upper filling mark, in tube having bulb at lower height; viscosity of liquid selected to be, in the measurement range of viscometer.

**B-2.3** Allow 20 min for viscometer to attain the test temperature. With the help of rubber below apply pressure to raise the liquid through capillary above the upper mark of the other bulb. Release the pressure and allow the liquid to flow through capillary. Measure time required for the liquid meniscus to fall from upper mark to lower mark of the bulb.

**B-2.4** Calculate viscometer constant,  $C$  of liquid using the following formula:

$$C = V/T$$

where

$V$  = viscosity, in cS; and

$T$  = time, in seconds.

### B-2.5 Acceptance Criteria

Ostwald U-tube viscometer is stated to be calibrated if viscometer constant is within  $\pm 10$  percent of the expected tube constant (see Table 7). If value exceeds this limit then estimated constant is to be used in the calculations of viscosity of the sample under test.

**Table 7 Acceptance Criteria**  
(Clause B-2.5)

Sl No.	Type of U-Tube Viscometer	Viscosity Range, cS	Expected Viscometer Constant, $C$
(1)	(2)	(3)	(4)
i)	A	1.5 - 10	0.003
ii)	B	4.0 - 10	0.01
iii)	C	7.5 - 35	0.03
iv)	D	20 - 120	0.1
v)	E	60 - 500	0.3
vi)	F	200 - 1 000	1.0
vii)	G	600 - 4 000	3.0
viii)	H	2 000 - 15 000	10.0

**ANNEX C**  
**( Clause 8.4.1.1.1)**

**C-1 APPARATUS**

**C-1.1 Standard Viscosity Fluids**

Recommended by supplier of instrument having viscosities at about 5 poise, 50 poise and 125 poise at 25°C or of a value that covers the working range of the resin sample. The standard fluids will have test certificate and assurance of stability of fluid material.

**C-1.2 Glass Beaker, 600 ml.**

**C-1.3** A calibrated thermometer of a range of -10° to 110°C with a least count of 0.5°C.

**C-2 PROCEDURE**

Condition the standard viscosity fluids and the instrument at 27±0.5°C. Determine the viscosity of standard fluid on Brookfield Viscometer using suitable spindle and RPM, as given in the operating guide of the instrument.

Calculate the viscosity by multiplying the reading with corresponding factor from factor finder. In case of instruments, where viscosity is displayed directly, the application of factor is not necessary. Report the viscosity in centipoise or poise.

**C-3 ACCEPTANCE CRITERIA**

The instrument is said to be calibrated when the observed value, of viscosity is within ±2 percent of the certified value of the standard fluids at 27°C.

( CHD 20 )